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Report 2462

Antioxidant Systems for Elastomeric Tank Pad Formulations

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This report investigates the utilization of various antioxidant systems—including blends of two or more antioxidants on various polymers—in an effort to improve the oxidative degradation of elastomeric tank pad compounds.

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PREFACE

This technical report, prepared by the Materials, Fuels and Lubricants Directorate of the US Army Belvoir Research, Development and Engineering (RD&E) Center, Fort Belvoir, VA, was sponsored and funded by the US Army Tank-Automotive Command (TACOM), Warren, MI, and the US Army Materials Technology Laboratory, Watertown, MA. It investigates the utilization of various antioxidant systems—including blends of two or more antioxidants on various polymers—in an effort to improve the oxidative degradation of elastomeric tank pad compounds.

Military tracked vehicles are equipped with rubber blocks or endless-band rubber track to reduce shock, noise, wear, and damage to road surfaces. Historically, field performance of the elastomeric components of such tracked vehicles as the M-60 and M-1 Army tanks has been poor. Current service life for the M-1 tank pads has been reported as low as 300 miles—significantly short of the 2,000 mile life expectancy. The problem is further complicated with off-the-road service conditions where pads fail at a much faster rate. The severity of the wear is more pronounced on the M-1 main battle tank than on the M-60 due to increased weight and speed with a smaller rubber footprint, which produces higher stresses and heat build-up. Therefore, costly and frequent replacement is necessary to keep the tanks operational.

Conventional track pads based on styrene-butadiene rubber (SBR) usually fail prematurely in service because of poor chunking and chipping resistance, excessive wear, blow-out or rubber-to-metal bond failure. Obviously, improved service durability of rubber track pads would provide economic, tactical and logistical advantages for modern, high speed tracked vehicles. This study on antioxidants points out the best protection system against oxidative degradation. RUBBER. (TES)

To improve the quality of tank pads, TACOM sponsored a series of investigations during Fiscal Year (FY) 1983 to 1987 involving industry, government, and academia. The Rubber and Coated Fabrics Research Group of the Belvoir RD&E Center conducted a series of studies between FY 1985 to 1987 to improve the service life of tank pads to include: processing variations, filler dispersion, type of polymer, blend of polymers, curing and filler systems, use of short fiber reinforcement, and the study of different antioxidant systems to improve heat resistance. This initial study was not geared to optimize the antioxidant systems but to gather information and create an extensive data base of available antioxidants. The information collected will be used at later stages in the program for studies on heat resistance optimization and tailoring compounds to specific performance characteristics.

CONTENTS

SECTION I. INVESTIGATION TECHNIQUES

OXIDATIVE STABILITY

The properties of polymers change with the passage of time, particularly at elevated temperatures. These changes may involve chain breakdown (chain scission), crosslink formation, and changes in the color characteristics of the polymer. For any given polymer or blend, the oxidative stability of the rubber compound is determined by:

- Service conditions
- Nature of the polymer
- Type of crosslinks
- Choice of antidegradant.

To counteract the deterioration of the elastomer, whether by natural or accelerated aging, it is necessary to add materials which are capable of retarding these types of deterioration. The proper cure system can play a very important role in providing good heat or oxidation resistance for general purpose elastomer systems. It is not within the scope of this report to review these cure systems in depth; reduced sulfur levels plus increased accelerator or sulfur donor will provide major improvements in heat and oxidation resistance.

The oxidative degradation processes have a common basis in the radical chain auto-oxidation mechanism originally proposed by Bolland and his co-workers.[1,2,3] The oxidation of elastomers and other organic materials with elemental oxygen is called *auto-oxidation* because it is an autocatalytic process in which hydroperoxides, formed as the primary product of the reaction, decompose to produce free radicals which initiate the free radical chain mechanism ultimately destroying the rubber.

R. + P ------------------------> ROO.

ROO. + RH --------------------------> ROOH + R.

The oxidation inhibition processes have been classified in two main types: kinetic chain-breaking processes and initiation prevention mechanisms. Antioxidants which retard the formation free radicals in the initiation step are referred to as *preventive antioxidants*, and those which interrupt the

propagation cycle by reacting with either R. or ROO. radicals are called chain-breaking antioxidants. The first type embraces the hydroperoxide decomposers, the traditional metal deactivators and u.v. stabilizers, while the second type includes the traditional rubber antioxidants, the aromatic amine, and phenols.

Oxidative aging generally causes a decrease in tensile, tear, and fatigue properties. The effect on stiffness depends on the formulation and the temperature of aging. Lower temperatures favor an increase in stiffness, particularly in the early stages. At high temperatures, oxidative aging affects only the surface since essentially all the oxygen is consumed by reaction with antioxidants or rubber before it can diffuse into the bulk. Generally, a resinous skin is formed at high temperatures, thus degradation in the center of a large component is essentially anaerobic. In tank pads, the flexing, cutting, and chipping actions occur at a faster rate than the formation of the resinous skin; therefore, extreme care must be used in interpreting aging data obtained from tests on thin strips when compared with tank pad performance. This type of resinous skin was observed when tensile and tear strength specimens were heat aged for 4 hours at 300°F.

Before we analyze antioxidants in more detail, it is very important to keep in mind the most common causes of elastomer degradation, which include:

- Reaction with oxygen
- Heat
- Light and weathering
- Metal ions (pre-oxidants)
- Dynamic fatigue
- Ozone.

COMPOUNDING FOR HEAT RESISTANCE

Even though many studies have been completed about the mechanism of oxidation of hydrocarbons, the vulcanization chemical reactions of ordinary elastomers is very complicated. With the possible exception of radiation and peroxides cures, the extensive mechanistic studies on oxidation are of little assistance to the rubber compounder. Since the various antioxidants function by different mechanisms, a material that acts as an antioxidant under one condition may become an oxidation promoter in a different condition. When used in combination, antioxidants sometimes behave synergistically; in other cases they can be antagonistic to each other. Furthermore, the level at which they are used is important since an increase beyond certain critical levels can decrease the

protection given, acting then as a pro-oxidant. It is also clear that the introduction of sulfur-containing cross-links and the by-products of vulcanization cause wide variations in the resistance to oxidation of the rubber. For these reasons, in order to optimize for heat resistance, the rubber compunder is forced to design experiments to select optimum levels and combinations of antioxidants for any given rubber formulation.

As mentioned earlier, some of the more important causes for degradation of tank pad rubbers are heat and dynamic fatigue. Obviously, heat accelerates oxidation and dynamic fatigue can develop cracks in the vulcanizate surface which, under fatigue conditions, will grow causing catastrophic failures. The selection of the proper antidegradant system for tank pads is not an easy task and some of their more important properties must be considered, such as:

- Volatility
- Solubility
- Stability
- Physical state
- Antidegradant concentration.

Due to the service conditions for tank pad applications, the groups of antioxidants given the most importance were the p-phenylenediamines (PPD), Quinolines (TMQ), and phenols (especially the ones with more than one benzene ring). The p-phenylenediamines are probably the most effective antioxidants against oxidation and flex cracking, while the phenols exhibit very low volatilities and therefore a high resistance to leaching as the temperature builds up on the tank pads. It is obvious that a volatile antioxidant will only have a limited period of usefulness in the rubber. Table 1 shows the melting point of several antioxidants used as an indication of volatility resistance upon aging.

Table 1. Antioxidant Melting and Boiling Points

ANTIOXIDANT COMMERCIAL NAME	MELTING POINT °C	BOILING POINT °C
Ethanox 330	244	
Ethanox 702	154	
Ethanox 744		298
Ethanox 701	36	
Ethyl Antioxidant 703	94	
Ethyl Anioxidant 736	124	
Flexamine G	95-115	
Flexzone 7L	50	
Flexzone 6H	110	
Flexzone 3C	74	
Naugard Q	75	
Naugard 445	96-99	
Naugard 477	95-100	
Naugard PAN	55	
Naugard BXA	85-95	
Octamine	75-85	
Permanax TQ	79	
Permanax WSP	130	
Permanax WSO	168	
Permanax WSL		171
Permanax CNS	298	
Permanax DPPD	130	
Permanax IPPD	70	
Santoflex 13	44-49.5	
Vanox MTI	250	
Vanox ZMTI	300	
Vanox 1290	161-164	
Vanox 25`	95	
Vulcanox 4010 NA	75	
Vulcanox MB-2/MG	290	
Vulcanox 4020	45	
Vulcanox		237
Wingstay 100	90-105	
Wingstay 300	45-50	
Agerite Hipar T	70	

Table 1. Antioxidant Melting and Boiling Points – Continued

ANTIOXIDANT COMMERCIAL NAME	MELTING POINT °C	BOILING POINT °C
Agerite Resin D	74	
Agerite White	224-230	
Agerite DPPD	144-152	
Agerite HPS	80-100	
Agerite MA	105	
Agerite SKT	123-131	
Agerite Stalite S	89-103	
Akrochem Antioxidant 2246	124	
Akrochem PD-1	75	
Akrochem PANA	50	
Akrochem Antioxidant PD-2	45	
Akrochem Antioxidant 58	300	
Akrochem Antioxidant DQ	75	
Akrochem Antioxidant S	88	
Akrochem Antioxidant 36	148	
Akrochem Antioxidant MB	290	
Akrochem Antioxidant ZMB	300	
Aminox 5664	85-95	
Antozite 67	40-44	

SECTION II. EXPERIMENTAL TESTING

APPROACH

Numerous antioxidants were compounded with natural, SBR, nitrile, and highly saturated nitrile rubbers as well as blends of the different polymers. However, the work detailed in this report was performed on natural rubber. Some of the antioxidant groups used in the study included:

- Hindered "Bis" phenols
- Organic sulfur compounds
- Mercaptotolylimidazole and its zinc salts
- Phenylnapthylamines
- Dihydroquinolines
- Diphenylamine derivatives
- Substituted paraphenylenediamines

The control compound used was Natural rubber (RSS-1)-100, zinc oxide-4, stearic acid-2, N-110 carbon black-45, Sulfur-2.5, and N-Cyclohexyl-2-benzothiazolesulfenamide (Santocure)-0.8. Six of the 95 compounds evaluated also contained 3phr of Antozite 2 (N, N'-di-3[5-methylheptyl]-p-phenylenediamine). Table 2 lists the chemical and commercial names of the antioxidants evaluated.

Three batches of rubber were mixed and vulcanized for each compound. Thus, specimens used in each test contain duplicates, since several were taken from each batch to provide the required number of specimens and to give a representative cross-section relative to reproducibility of processing and test results. All compounds were cured under the same conditions of time and temperature as determined from rheometer curves.

It has been reported[4] that at an average speed of 20 mph and temperatures of up to 260°F were observed on the T-142 rubber pads of the M-60 tank. Based on these findings, heat aging at 212°F and 250°F were selected as the critical temperatures for evaluation of tank pad materials.

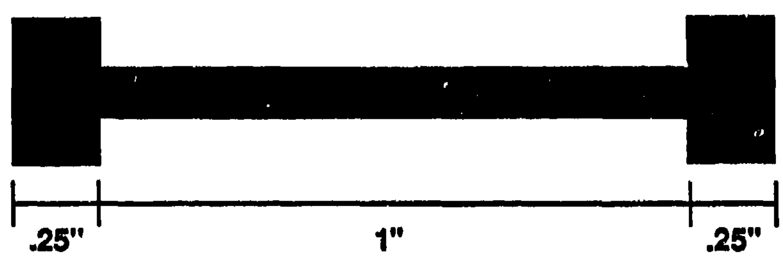
Table 2. Antioxidant Chemical and Commercial Names

CHEMICAL NAMES	COMMERCIAL NAME
1. N, N'-Bis-(1 ethyl-3-methyl pentyl)-p-phenylenediamine	Antozite2
2. N, N'-Di-beta-naphthyl-p-phenylenediamine	Agerite White
3. Polymerized 1, 2-dihydro-2, 2, 4-trimethylquinoline	Agerite Resin D, Agerite MA, Naugard Q, Akrochem Antioxidant DQ, Permanax TQ
4. 50% Phenyl-beta-naphthylamine, 25% DPPD, 25%-p-Isopropoxydiphenylamine	Agerite Hipar
5. High temperature reaction product of diphenylamine and acetone (structure not disclosed), Naugard BXA includes also reaction with formaldehyde	Agerite Superflex & Superflex Solid G, BLE 75 (75% active), Permanax BL, BLE 25, Naugard BXA
6. 6-Ethoxy-1, 2-dihydro-2, 2, 4,-trimethyl-quinoline	Santoflex AW
7. 50% Alkylated diphenylamines (Agerite Stalite S), 25% High temperature reaction product of diphenylamine and acetone	Agerite Hipar T
8. Mixed ditolyl PPD (undisclosed structure)	Wingstay 100
9. N-1, 3-dimethylbutyl-N'-phenyl-p-phenylenediamine	Wingstay 300, Akrochem Antioxidant PD-2, Santoflex 13, Antozite 67, Vulcanox 4020, Flexzone 7L
10. Octylated diphenylamine (structure not disclosed)	Octamine, Agerite Stalite, Agerite Stalite S, Akrochem Antioxidant S
11. 2-2'-methylene-bis-4methyl-6-t-butyl phenol	Akrochem Antioxidant 2246
12. N-isopropyl-N'-phenyl-p-phenylene diamine	Akrochem Antioxidant PD-1, Permanax IPPD, Vulcanox 4010 NA, Flexzone 3C
13. Phenyl-alpha-naphthylamine	Akrochem Antioxidant PANA, Naugard PAN
14. N, N'-Dyphenyl-p-phenylenediamine	Agerite DPPD, Permanax DPPD
15. 65% Diarylamine-ketone reaction product 35% DPPD	Flexamine G
16. 65% Alkylated diphenylamines, 35% DPPD	Agerite HP-S
17. Phenyl-beta naphthylamine	Additin 30
18. 2-Mercaptobenzimidazole	Akrochem Antioxidant MB, Vulcanox MB-2 MG
19. Zinc-2-mercaptobenzimidazole	Akrochem Antioxidant ZMB
20. 2-Mercaptotolylimidazole	Vanox MTI
21. 2, 6-Di-t-butyl-alpha-dimenthyl amino p-cresol	Ethanox 703

Table 2. Antioxidant Chemical and Commercial Names—Continued

CHEMICAL NAMES	COMMERCIAL NAME
22. 4, 4'-Thio-bis-(2-methyl-6-t-butyl phenol)	Ethanox 736
23. 3, 5-Di-t-Butyl-4Hydroxyhydrocinnamic Acid Triester with 1, 3, 5-Tris (2-Hydroxy-Ethyl)-s-Triazine-2, 4, 6-(1H, 3H, 5H) Trione	Agerite SKT
24. Zinc 2-Mercaptotolylimidazole	Akrochem Antioxidant 58, Vanox ZMTI
25. Polymeric phenol	Akrochem Antioxidant 36
26. 2, 2'-Methylenebis (4-methyl-6-(1-methyl-cyclohexyl) phenol)	Permanax WSP
27. Liquid Alkylated bis-phenols (structure not disclosed)	Permanax WSO
28. Alkylated phenols (structure not disclosed)	Permanax WSL
29. Blend of Alkyl and Aryl p-phenylene diamines	Flexzone 11L & 12L
30. N-Cyclohexyl-N'-phenyl-p-phenylene-daimine	Flexzone 6H
31. N, N'-Bis-(1, 4-dimethylpentyl)-p-phenylenediamine	Vulcanox 4030, Flexzone 4L
32. PPD Blends	Santoflex 134D, Flexzone 15L
33. 4, 4'-Methylene-bis-(2, 6-di-t-butyl phenol)	Ethanox 702
34. 2, 6-ditert-butyl phenol	Ethanox 701
35. 1, 3, 5-Trimethyl-2, 4, 6-Tris (3, 5-Di-t-Butyl-4-Hydroxybenzyl)-Benzene	Ethanox 330
36. Styrenated phenol (mixture of mono, di, tri)	Vanox 102
37. 2, 2'-Ethylidenebis (4, 6-di-t-butylphenol)	Vanox 1290
38. 6-dodecyl-1, 2-dihydro-2, 2, 4-tri-methylquinoline	Santoflex DD

All tensile strength specimens were prepared according to ASTM D-412, *Test Methods for Rubber Properties in Tension,* using Die C specimens. The results reported in Table 3 are the average of three specimens. The tensile strength determinations for 212°F and 250°F were conducted at room temperature after aging for 70 hours at the elevated temperatures. The specimens used for the stress relaxation test were of the same type of those described Type B specimen in ASTM D-1053, *Test Method for Rubber Property—Stiffening at Low Temperature: Flexible Polymers and Coated Fabrics*. The figure below illustrates the Type B specimen dimensions. The specimens for the differential scanning calorimetry work were prepared by cutting small portions of rubber from a 6 by 6 inch standard vulcanized sheet as described in ASTM D-3182, *Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets*. The rubber was then weighted to approximately 20 mg. To prevent wide thermal variations, hermetically sealed sample pans were used.



EVALUATION OF ANTIOXIDANTS

The evaluation of antioxidants is a very difficult process since many variables can affect the results and confuse the issue. For example, retained tensile strength is often considered one valuable test, but in many cases shows either no effect at all or an opposite effect to that expected. This is true particularly in SBR or BR, while tensile is more reliable in natural rubber. In this study, both properties (tensile strength and elongation) were measured to eliminate any evaluation bias. The tensile strength and elongation retention were measured after 70 hours heat aging at 212°F and 250°F. The product of tensile strength and elongation is shown in Table 4 to highlight the effect of aging for the different antioxidant systems. Table 5 provides conversion factors to convert results to SI.

In most rubber formulations and under most circumstances, the elongation of rubber decreases faster during aging than its strength. Thus elongation is a critical parameter and the rate of its decrease determines the useful life of rubber at a given temperature.[5] Consequently, the loss of elongation may be a more sensitive criterion for aging measurements regardless of mechanism, over tensile loss for cured compounds. However, since the work detailed in this report deals only with natural rubber, retention of tensile strength and elongation after aging at 250°F is used as the main criteria to rank antioxidant efficiency. Consideration is given to the level of the original tensile strength along with the retention upon aging. Tables 3 and 6 illustrate the ranking on all compounds tested.

Table 3. Effect of Antioxidants on Physical Properties

FORMULATION ID	COMMERCIAL NAME	ANTIOXIDANT TYPE	TENSILE STRENGTH TENSILE (PSI)
A	None		3767
B	Antozite2	Dialkyl PPD	3750
C	Agerite White	Diaryl PPD	3408
D	Agerite Resin D	Quinoline	3683
E	Agerite Resin D/White	Quinoline/Diaryl PPD	3083
F	Agerite Resin D/White/Antozite2/Agemaster	Quinoline/Diaryl PPD/Dialkyl PPD	3733
G	Agemaster		3033
H	Agerite Hipar/Antozite2	Blended Amine/Dialkyl PPD	3700
I	Santoflex AW/Antozite2	Quinoline/Dialkyl PPD	3867
J	Agerite Superflex Solid G/Antozite2	Carbonyl-amine Condensation Prod/Dialkyl PPD	3625
K	Agerite Hipar T/Santoflex AW/Antozite2	Cond Prod/Quinoline/Dialkyl PPD	3575
L	Agerite White/Super Solid G/Antozite2	Cond Prod/Dialkyl PPD/Diaryl PPD	3767
M	Wingstay 300/Antozite2	Alkyl-Aryl PPD/Dialkyl PPD	3733
N	Wingstay 100	Mixed Ditolyl PPD	3750
O	Octamine	Diphenylamine	3917
P	Akrochem Antioxidant 2246	Hindered Bisphenol	3783
Q	Akrochem Antioxidant PD-1	Alkyl-Aryl PPD	3983
R	Akrochem Antioxidant PANA	Phenyl-a-Naphthylamine	3767
S	Agerite DPPD	Diaryl PPD	3747
T	Flexamine G	Blended Amines	3917
U	Agerite HPS	Blended Amines	3758
V	Akrochem Antioxidant PD-2/Santoflex 13	Alkyl-Aryl PPD	3687
W	Additin 30	Naphthylamine	3910
X	BLE 75	Carbonyl-amine Condensation Product	3883
Y	Antioxidant MB (Mobay)	Mercaptobenzimidazole, MBI	3787
Z	Antioxidant ZMB (Mobay)	Mercaptobenzimidazole Zinc salt, ZMBI	3673
AA	Vanox MTI	Mercaptotolylimidazole, MTI	3607
BB	Agerite MA	Quinoline	3447
CC	Ethyl Antioxidant 703	Hindered Bisphenol	3370
DD	Ethyl Antioxidant 736	Hindered Bisphenol	3300
EE	Antioxidant MB/Agerite Resin D	Mercaptobenzimidazole/Quinoline	3290
FF	Antioxidant ZMB/Agerite Resin D	Mercaptobenzimidazole Zinc salt/Quinoline	3240
GG	Vanox MTI/Agerite Resin D	Mercaptotolylidimazole/Quinoline	3200
HH	Agerite SKT	miscellaneous	3537
II	Agerite Stalite S	Diphenylamine	3807
JJ	Agerite Stalite	Diphenylamine	3690
KK	Antozite 67	Alky-Aryl PPD	3887
LL	Agerite Resin D/Santoflex AW	Alkyl-Aryl PPD/Quinoline	3327
MM	Santoflex 13	Alkyl-Aryl PPD	3904
NN	Akrochem Antioxidant 58	Mercaptotolylimidazole Zinc salt, ZMI	3940
OO	Akrochem Antioxidant DQ	Quinoline	3977
PP	Akrochem Antioxidant S	Diphenylamine	3943
QQ	Akrochem Antioxidant 36	Hindered Phenol	3597
RR	Permanax TQ	Quinoline	3693
SS	Permanax BL	Carbonyl Amine Condensation Product	3783
TT	Permanax WSP	Hindered Bisphenol	3807
UU	Permanax WSO	Hindered Bisphenol	4323
VV	Permanax WSL	Hindered Phenol	3857
WW	Permanax CNS	Blend of Non-staining Antioxidants	3740
XX	Permanax DPPD	Diaryl PPD	3777
YY	Permanax IPPD	Alkyl-Aryl PPD	3860
ZZ	Vulcanox 4010 NA	Alkyl-Aryl PPD	3787
AX	Vulcanox MB-2/MG	Mercaptotolylimidazole, MTI	3630
BX	Vulcanox 4020	Alkyl-Aryl PPD	3723
CX	Naugard Q	Quinoline	3655
DX	Naugard 445	Diphenylamine	3750
EX	Naugard 447	Blended Amines	3750

Table 3. Effect of Antioxidants on Physical Properties--Continued

FORMULATION ID	COMMERCIAL NAME	ANTIOXIDANT TYPE	TENSILE STRENGTH TENSILE (PSI)
FX	Naugard PAN	Naphthylamine	3770
GX	Naugard BXA	Rx Prod of Diphenylamine, acetone & formaldehyde	3600
HX	Naugard Q/Naugard 445	Quinoline/Diphenylamine	3810
IX	Naugard BXA/Naugard Q	Quinoline/Hindered Phenol	3933
JX	Flexzone 12L	Blend of PPD	3675
KX	Flexzone 7L	Alkyl-Aryl PPD	3870
LX	Vulcanox 4030	Dialkyl PPD	3887
MX	Vanox ZMTI	Mercaptotolylimidazole Zinc salt, ZMTI	3408
OX	Permanex BL	Caronyl-amine Condensation Product	4067
PX	Flexzone 15L	Blend of PPD	4150
QX	Flexzone 6H	Alkyl-Aryl PPD	4200
RX	Flexzone 3C	Alkyl-Aryl PPD	3817
SX	Flexzone 4L	Dialkyl PPD	3900
TX	Flexzone 11L	Blend of PPD	3750
UX	Flexzone HS		4100
VX	Akrochem Antioxidant 58	Mercaptotolylimidazole Zinc salt, ZMTI	3867
WX	Aminox 5664	Condensation Products	3967
XX2	Ethanox 330	miscellaneous	4183
ZX	Ethanox 702	Hindered Bisphenol	3950
AAX	Ethanox 744		4283
BBX	Ethanox 701	Hindered Phenol	4250
CCX	Antozite 67	Alkyl-Aryl PPD	3825
DDX	BLE 25/Flexamine G	Carbonyl-amine Cond Prod/Blended Amine	3633
EEX	Santoflex 13	Alkyl-Aryl PPD	3767
FFX	Santoflex 13/Agerite Resin D	Alkyl-Aryl PPD/Quinoline	3950
GGX	Santoflex 13/Agerite MA	Alkyl-Aryl PPD/Quinoline	2667
HHX	Agerite Resin D/Santoflex/Vanox MTI	Alkyl-Aryl PPD/Quinoline/MTI	4183
IIX	Agerite MA/Santoflex 13/Vanox MTI	Alkyl-Aryl PPD/Quinoline/MTI	1983
JJX	Agerite Superflex Solid G/Resin D/Vanox MTI	Cond Prod/Quinoline/MTI	2217
KKX	Agerite Stalite S/Resin D/Vanox MTI	Diphenylamine/Quinoline/MTI	3683
LLX	Vanox 1320/Santoflex/Agerite Resin D	Alkyl-Aryl PPD/Quinoline/Hindered Bisphenol	2383
MMX	Santoflex 134D/Agerite Resin D/Vanox 102	Alky-Aryl PPD/Quinoline/Hindered Phenol	2200
NNX	Santoflex 134D/Agerite Resin D/Vanox 1290	Alkyl-Aryl PPD/Quinoline/Hindered Bisphenol	2283
OOX	Santoflex 134D/Agerite Resin D/Vanox ZS	Alkyl-Aryl PPD/Quinoline/Hindered Phenol	1850
PPX	Santoflex 134D/Agerite Resin D/Santoflex DD	Alkyl-Aryl PPD/Quinoline/Quinoline	2000
QQX	Santoflex 134D/Agerite Resin D/Vanox MTI	Alkyl-Aryl PPD/Quinoline/MTI	1567
RRX	Santoflex 134D/Agerite Resin D/Santoflex 13	Alkyl-Aryl PPD/Quinoline/Alkyl-Aryl PPD	3967
SSX	Santoflex 13/Agerite Resin D/DTDTDP	Alkyl-Aryl PPD/Quinoline	4183

Table 3. Effect of Antioxidants on Physical Properties–Continued

FORMULATION ID	COMMERCIAL NAME	TENSILE STRENGTH @212°F (PSI)	TENSILE STRENGTH @250°F (PSI)	ULTIMATE ELONGATION ORIGINAL (%)	ULTIMATE ELONGATION @212°F (%)	ULTIMATE ELONGATION @250°F (%)
A	None	1550	442	457	210	43
B	Antozite2	2173	467	480	263	53
C	Agerite White	1917	500	433	247	43
D	Agerite Resin D	2540	558	457	283	67
E	Agerite Resin D/White	2300	467	447	290	50
F	Agerite Resin D/White/Antozite2/Agemaster	3650	993	567	517	160
G	Agemaster	3033	707	427	427	103
H	Agerite Hipar/Antozite2	2753	637	493	330	87
I	Santoflex AW/Antozite2	2650	510	510	317	60
J	Agerite Superflex Solid G/Antozite2	2530	550	510	330	83
K	Agerite Hiper T/Santoflex AW/Antozite2	2453	590	500	323	87
L	Agerite White/Super Solid G/Antozite2	2453	620	510	323	83
M	Wingstay 300/Antozite2	2023	540	513	287	67
N	Wingstay 100	1737	533	480	233	60
O	Octamine	1497	590	477	237	87
P	Akrochem Antioxidant 2246	1136	500	477	193	67
Q	Akrochem Antioxidant PD-1	1564	530	483	223	67
R	Akrochem Antioxidant PANA	1170	457	480	217	60
S	Agerite DPPD	1988	643	450	247	80
T	Flexamine G	1720	600	477	230	77
U	Agerite HPS	1628	610	460	220	80
V	Akrochem Antioxidant PD-2/Santoflex 13	1800	303	510	243	73
W	Additin 30	2110	337	513	253	73
X	BLE 75	2243	293	527	273	70
Y	Antioxidant MB (Mobay)	3033	553	550	387	127
Z	Antioxidant ZMB (Mobay)	2703	387	517	333	97
AA	Vanox MTI	2317	497	540	383	120
BB	Agerite MA	2390	503	507	297	83
CC	Ethyl Antioxidant 703	2453	457	477	297	77
DD	Ethyl Antioxidant 736	1870	413	503	250	70
EE	Antioxidant MB/Agerite Resin D	2297	600	557	387	120
FF	Antioxidant ZMB/Agerite Resin D	2760	633	537	340	113
GG	Vanox MTI/Agerite Resin D	3033	703	433	363	133
HH	Agerite SKT	2447	433	510	320	63
II	Agerite Stalite S	2590	517	493	323	80
JJ	Agerite Stalite	2727	537	497	337	83
KK	Antozite 67	2597	483	520	313	73
LL	Agerite Resin D/Santoflex	3200	933	583	450	143
MM	Santoflex 13	2433	533	523	303	73
NN	Akrochem Antioxidant 58	2847	533	517	350	77
OO	Akrochem Antioxidant DQ	2270	493	503	290	63
PP	Akrochem Antioxidant S	2333	560	500	310	77
QQ	Akrochem Antioxidant 36	2067	517	483	283	70
RR	Permanax TQ	2287	505	487	313	83
SS	Permanax BL	2427	497	517	330	77
TT	Permanax WSP	1853	527	497	270	80
UU	Permanax WSO	1770	522	510	263	73
VV	Permanax WSL	2100	457	490	287	70
WW	Permanax CNS	2150	503	530	303	90
XX	Permanax DPPD	2180	500	493	317	60
YY	Permanax IPPD	2240	463	520	313	77
ZZ	Vulcanox 4010 NA	2373	513	527	300	83
AX	Vulcanox MB-2/MG	2977	547	540	387	120
BX	Vulcanox 4020	2520	508	510	323	80
CX	Naugard Q	2777	617	500	337	80
DX	Naugard 445	2597	637	500	323	53
EX	Naugard 477	2570	542	500	320	73

Table 3. Effect of Antioxidants on Physical Properties–Continued

FORMULATION ID	COMMERCIAL NAME	TENSILE STRENGTH @212°F (PSI)	TENSILE STRENGTH @250°F (PSI)	ULTIMATE ELONGATION ORIGINAL (%)	ULTIMATE ELONGATION @212°F (%)	ULTIMATE ELONGATION @250°F (%)
FX	Naugard PAN	2733	598	490	317	77
GX	Naugard BXA	2280	637	510	347	77
HX	Naugard Q/Naugard 445	2613	580	540	323	73
IX	Naugard BXA/Naugard Q	2550	553	537	320	63
JX	Flexzone 12L	2630	670	540	287	93
KX	Flexzone 7L	2623	597	520	320	73
LX	Vulcanox 4030	2677	600	530	347	90
MX	Vanox ZMTI	2958	621	537	405	90
OX	Permanax BL	2887	698	520	377	87
PX	Flexzone 15L	3080	613	540	403	80
QX	Flexzone 6H	2980	657	537	387	77
RX	Flexzone 3C	2657	653	550	353	87
SX	Flexzone 4L	2797	617	497	377	80
TX	Flexzone 11L	2727	585	497	387	77
UX	Flexzone HS	2793	648	517	367	77
VX	Akrochem Antioxidant 58	3373	783	540	473	123
WX	Aminox 5664	2900	797	573	397	80
XX2	Ethanox 330	2430	703	523	337	87
ZX	Ethanox 702	3577	653	560	360	80
AAX	Ethanox 744	3065	697	527	420	110
BBX	Ethanox 701	2630	690	560	393	90
CCX	Antozite 67	2983	592	533	350	67
DDX	BLE 25/Flexamine G	2892	775	560	433	98
EEX	Santoflex 13	2263	653	530	413	103
FFX	Santoflex 13/Agerite Resin D	2650	650	543	463	90
GGX	Santoflex 13/Agerite MA	2327	757	533	410	103
HHX	Agerite Resin D/Santoflex 13/Vanox MTI	2573	753	573	370	90
IIX	Agerite MA/Santoflex 13/Vanox MTI	2607	803	503	453	130
JJX	Agerite Superflex Solid G/Resin D/Vanox MTI	2793	790	480	500	123
KKX	Agerite Stalite S/Resin D/Vanox MTI	3313	1018	597	510	167
LLX	Vanox 1320/Santoflex 13/Agerite Resin D	2120	794	487	393	113
MMX	Santoflex 134D/Agerite Resin D/Vanox 102	2120	767	460	373	97
NNX	Santoflex 134D/Agerite Resin D/Vanox 1290	2117	765	470	370	97
OOX	Santoflex 134D/Agerite Resin D/Vanox ZS	2353	773	417	397	100
PPX	Santoflex 134D/Agerite Resin D/Santoflex DD	1963	733	470	387	107
QQX	Santoflex 134D/Agerite Resin D/Vanox MTI	2750	838	417	503	140
RRX	Santoflex 134D/Agerite Resin D/Santoflex 13	2690	813	510	400	97
SSX	Santoflex 13/Agerite Resin D/DTDTDP	2633	787	523	363	97

Table 4. Retention of Physical Properties After Aging at 250°F

FORMULATION ID	COMMERCIAL NAME	ORIGINAL TENSILE (PSI)	TENSILE @250°F (PSI)	ELONG RET @250°F (%)	TEN RET @250°F (%)	TEN*ELONG @250°F X 1000
A	None	3767	442	43	11.7	19.01
QQX	Santoflex 134D/Agerite Resin D/Vanox MTI	1567	838	140	53.5	117.32
OOX	Santoflex 134D/Agerite Resin D/Vanox ZS	1850	773	100	41.8	77.30
IIX	Agerite MA/Santoflex 13/Vanox MTI	1983	803	130	40.5	104.39
PPX	Santoflex 134D/Agerite Resin D/Santoflex DD	2000	733	107	36.7	78.43
JJX	Agerite Superflex Solid G/Resin D/Vanox MTI	2217	790	123	35.6	97.17
MMX	Santoflex 134D/Agerite Resin D/Vanox 102	2200	767	97	34.9	74.40
NNX	Santoflex 134D/Agerite Resin D/Vanox 1290	2283	765	97	33.5	74.21
LLX	Vanox 1320/Santoflex 13/Agerite Resin D	2383	794	113	33.3	89.72
GGX	Santoflex 13/Agerite MA	2667	757	103	28.	77.97
LL	Agerite Resin D/Santoflex	3327	933	143	28.	133.42
KKX	Agerite Stalite S/Resin D/Vanox MTI	3683	1018	167	27.6	170.01
F	Agerite Resin D/White/Antozite2/Agemaster	3733	993	160	26.6	158.88
G	Agemaster	3033	707	103	23.3	72.82
GG	Vanox MTI/Agerite Resin D	3200	703	133	22.0	93.50
DDX	BLE 25/Flexamine G	3633	775	98	21.3	75.95
RRX	Santoflex 134D/Agerite Resin D/Santoflex 13	3967	813	97	20.5	78.86
VX	Akrochem Antioxidant 58	3867	783	123	20.2	96.31
WX	Aminox 5664	3967	797	80	20.1	63.76
FF	Antioxidant ZMB/Agerite Resin D	3240	633	113	19.5	71.53
SSX	Santoflex 13/Agerite Resin D/DTDTDP	4183	787	97	18.8	76.34
EE	Antioxidant MB/Agerite Resin D	3290	600	120	18.2	72.00
JX	Flexzone 12L	3675	670	93	18.2	62.31
MX	Vanox ZMTI	3408	621	90	18.2	55.89
HHX	Agerite Resin D/Santoflex 13/Vanox MTI	4183	753	90	18.0	67.77
GX	Naugard BXA	3600	637	77	17.7	49.05
EEX	Santoflex 13	3767	653	103	17.3	67.26
H	Agerite Hipar/Antozite2	3700	637	87	17.2	55.42
OX	Permanax BL	4067	698	87	17.2	60.73
S	Agerite DPPD	3747	643	80	17.2	51.44
RX	Flexzone 3C	3817	653	87	17.1	56.81
DX	Naugard 445	3750	637	53	17.0	33.76
CX	Naugard Q	3655	617	80	16.9	49.36
XX2	Ethonox 330	4183	703	87	16.8	61.16
ZX	Ethonox 702	3950	653	80	16.5	52.24
K	Agerite Hipar T/Santoflex AW/Antozite2	3575	590	87	16.5	51.33
L	Agerite White/Super Solid G/Antozite2	3767	620	83	16.5	51.46
FFX	Santoflex 13/Agerite Resin D	3950	650	90	16.5	58.50
AAX	Ethanox 744	4283	697	110	16.3	76.67
BBX	Ethanox 701	4250	690	90	16.2	62.10
U	Agerite HPS	3758	610	80	16.2	48.80
FX	Naugard PAN	3770	598	77	15.9	46.05
SX	Flexzone 4L	3900	617	80	15.8	49.36
UX	Flexzone HS	4100	648	77	15.8	49.90
QX	Flexzone 6H	4200	657	77	15.6	50.59
TX	Flexzone 11L	3750	585	77	15.6	45.05
CCX	Antozite 67	3825	592	67	15.5	39.66
LX	Vulcanox 4030	3887	600	90	15.4	54.00
KX	Flexzone 7L	3870	597	73	15.4	43.58
T	Flexamine G	3917	600	77	15.3	46.20
HX	Naugard Q/Naugard 445	3810	580	73	15.2	42.34
J	Agerite Superflex Solid G/Antozite2	3625	550	83	15.2	45.65
D	Agerite Resin D	3683	558	67	15.2	37.39
E	Agerite Resin D/White	3083	467	50	15.1	23.35
AX	Vulcanox MB-2/MG	3630	547	120	15.1	65.64
O	Octamine	3917	590	87	15.1	51.33
PX	Flexzone 15L	4150	613	80	14.8	49.04

Table 4. Retention of Physical Properties After Aging at 250°F–Continued

FORMULATION ID	COMMERCIAL NAME	ORIGINAL TENSILE (PSI)	TENSILE @250°F (PSI)	ELONG RET @250°F (%)	TEN RET @250°F (%)	TEN*ELONG @250°F X 1000
C	Agerite White	3408	500	43	14.7	21.50
Y	Antioxidant MB (Mobay)	3787	553	127	14.6	70.23
BB	Agerite MA	3447	503	83	14.6	41.75
JJ	Agerite Stalite	3690	537	83	14.6	44.57
M	Wingstay 300/Antozite2	3733	540	67	14.5	36.18
EX	Naugard 477	3750	542	73	14.5	39.57
QQ	Akrochem Antioxidant 36	3597	517	70	14.4	36.19
N	Wingstay 100	3750	533	60	14.2	31.98
PP	Akrochem Antioxidant S	3943	560	77	14.2	43.12
IX	Naugard BXA/Naugard Q	3933	553	63	14.1	34.84
TT	Permanax WSP	3807	527	80	13.8	42.16
AA	Vanox MTI	3607	497	120	13.8	59.64
RR	Permanax TQ	3693	505	83	13.7	41.92
MM	Santoflex 13	3904	533	73	13.7	38.91
BX	Vulcanox 4020	3723	508	80	13.6	40.64
II	Agerite Stalite S	3807	517	80	13.6	41.36
CC	Ethyl Antioxidant 703	3370	457	77	13.6	35.19
ZZ	Vulcanox 4010 NA	3787	513	83	13.5	42.58
NN	Akrochem Antioxidant 58	3940	533	77	13.5	41.04
WW	Permanax CNS	3740	503	90	13.4	45.27
Q	Akrochem Antioxidant PD-1	3983	530	67	13.3	35.51
XX	Permanax DPPD	3777	500	60	13.2	30.00
P	Akrochem Antioxidant 2246	3783	500	67	13.2	33.50
I	Santoflex AW/Antozite2	3867	510	60	13.2	30.60
SS	Permanax BL	3783	497	77	13.1	38.27
DD	Ethyl Antioxidant 736	3300	413	70	12.5	28.91
B	Antozite2	3750	467	53	12.5	24.75
KK	Antozite 67	3887	483	73	12.4	35.26
OO	Akrochem Antioxidant DQ	3977	493	63	12.4	31.6
HH	Agerite SKT	3537	433	63	12.2	27.28
R	Akrochem Antioxidant PANA	3767	457	60	12.1	27.42
UU	Permanax WSO	4323	522	73	12.1	38.11
YY	Permanax IPPD	3860	463	77	12.0	35.65
VV	Permanax WSL	3857	457	70	11.8	31.99
Z	Antioxidant ZMB (Mobay)	3673	387	97	10.5	37.54
W	Additin 30	3910	337	73	8.6	24.60
V	Akrochem Antioxidant PD-2/Santoflex 13	3687	303	73	8.2	22.12
X	BLE 75	3883	293	70	7.5	20.51

Table 5. Conversion Table

U.S.	to	SI
1 $lb/in.^2$	=	6.894757 kPa
lb (Avoir)	=	0.4536 kg
°F	=	9/5(° + 32)
sq in. ($in.^2$)	=	6.4516 cm^2
lb/in.	=	175.1268 N/m
in.	=	25.4 millimeters
mil	=	.0254 millimeters

Table 6. Physical Properties by Antioxidant Type

FORMULATION ID	ANTIOXIDANT TYPE	TENSILE STRENGTH ORIGINAL (PSI)	TENSILE STRENGTH @212°F (PSI)	TENSILE STRENGTH @250°F (PSI)	ULTIMATE ELONGATION ORIGINAL (%)	ULTIMATE ELONGATION @212F°F (%)	ULTIMATE ELONGATION @250F°F (%)
A		3767	1550	442	457	210	43
AAX		4283	3065	697	527	420	110
UX		4100	2793	648	517	367	77
G		3033	3033	707	427	427	103
QX	Alkyl-Aryl PPD	4200	2980	657	537	387	77
Q	Alkyl-Aryl PPD	3983	1564	530	483	223	67
MM	Alkyl-Aryl PPD	3904	2433	533	523	303	73
KK	Alkyl-Aryl PPD	3887	2597	483	520	313	73
KX	Alkyl-Aryl PPD	3870	2623	597	520	320	73
YY	Alkyl-Aryl PPD	3860	2240	463	520	313	77
CCX	Alkyl-Aryl PPD	3825	2983	592	533	350	67
RX	Alkyl-Aryl PPD	3817	2657	653	550	353	87
ZZ	Alkyl-Aryl PPD	3787	2373	513	527	300	83
EEX	Alkyl-Aryl PPD	3767	2263	653	530	413	103
BX	Alkyl-Aryl PPD	3723	2520	508	510	323	80
V	Alkyl-Aryl PPD	3687	1800	303	510	243	73
M	Alkyl-Aryl PPD/Dialkyl PPD	3733	2023	540	513	287	67
SSX	Alkyl-Aryl PPD/Quinoline	4183	2633	787	523	363	97
FFX	Alkyl-Aryl PPD/Quinoline	3950	2650	650	543	463	90
LL	Alkyl-Aryl PPD/Quinoline	3327	3200	933	583	450	143
GGX	Alkyl-Aryl PPD/Quinoline	2667	2327	757	533	410	103
RRX	Alkyl-Aryl PPD/Quinoline/Alkyl-Aryl PPD	3967	2690	813	510	400	97
LLX	Alkyl-Aryl PPD/Quinoline/Hindered Bisphenol	2383	2120	794	487	393	113
NNX	Alkyl-Aryl PPD/Quinoline/Hindered Bisphenol	2283	2117	765	470	370	97
MMX	Alkyl-Aryl PPD/Quinoline/Hindered Phenol	2200	2120	767	460	373	97
OOX	Alkyl-Aryl PPD/Quinoline/Hindered Phenol	1850	2353	773	417	397	100
HHX	Alkyl-Aryl PPD/Quinoline/MTI	4183	2573	753	573	370	90
IIX	Alkyl-Aryl PPD/Quinoline/MTI	1983	2607	803	503	453	130
QQX	Alkyl-Aryl PPD/Quinoline/MTI	1567	2750	838	417	503	140
PPX	Alkyl-Aryl PPD/Quinoline/Quinoline	2000	1963	733	470	387	107
WW	Blend of Non-staining Antioxidants	3740	2150	503	530	303	90
PX	Blend of PPD	4150	3080	613	540	403	80
TX	Blend of PPD	3750	2727	585	497	387	77
JX	Blend of PPD	3675	2630	670	540	287	93
T	Blended Amines	3917	1720	600	477	230	77
U	Blended Amines	3758	1628	610	460	220	80
EX	Blended Amines	3750	2570	542	500	320	73
H	Blended Amine/Dialkyl PPD	3700	2753	637	493	330	87
SS	Carbonyl Amine Condensation Product	3783	2427	497	517	330	77
DDX	Carbonyl-amine Cond Prod/Blended Amine	3633	2892	775	560	433	98
OX	Carbonyl-amine Condensation Product	4067	2887	698	520	377	87
X	Carbonyl-amine Condensation Product	3883	2243	293	527	273	70
J	Carbonyl-amine Condensation Prod/Dialkyl PPD	3625	2530	550	510	320	83
L	Cond Prod/Dialkyl PPD/Diaryl PPD	3767	2453	620	510	323	83
K	Cond Prod/Quinoline/Dialkyl PPD	3575	2453	590	500	323	87
JJX	Cond Prod/Quinoline/MTI	2217	2793	790	480	500	123
WX	Condensation Products	3967	2900	797	573	397	80
SX	Dialkyl PPD	3900	2797	617	497	377	80
LX	Dialkyl PPD	3887	2677	600	530	347	90
B	Dialkyl PPD	3750	2173	467	480	263	53
XX	Diaryl PPD	3777	2180	500	493	317	60
S	Diaryl PPD	3747	1988	643	450	247	80
C	Diaryl PPD	3408	1917	500	433	247	43
PP	Diphenylamine	3943	2333	560	500	310	77
O	Diphenylamine	3917	1497	590	477	237	87
II	Diphenylamine	3807	2590	517	493	323	80
DX	Diphenylamine	3750	2597	637	500	323	53

Table 6. Physical Properties by Antioxidant Type—Continued

FORMULATION ID	ANTIOXIDANT TYPE	TENSILE STRENGTH ORIGINAL (PSI)	TENSILE STRENGTH @212°F (PSI)	TENSILE STRENGTH @250°F (PSI)	ULTIMATE ELONGATION ORIGINAL (%)	ULTIMATE ELONGATION @212°F (%)	ULTIMATE ELONGATION @250°F (%)
JJ	Diphenylamine	3690	2727	537	497	337	83
KKX	Diphenylamine/Quinoline/MTI	3683	3313	1018	597	510	167
UU	Hindered Bisphenol	4323	1770	522	510	263	73
ZX	Hindered Bisphenol	3950	3577	653	560	360	80
TT	Hindered Bisphenol	3807	1853	527	497	270	80
P	Hindered Bisphenol	3783	1136	500	477	193	67
CC	Hindered Bisphenol	3370	2453	457	477	297	77
DD	Hindered Bisphenol	3300	1870	413	503	250	70
BBX	Hindered Phenol	4250	2630	690	560	393	90
VV	Hindered Phenol	3857	2100	457	490	287	70
QQ	Hindered Phenol	3597	2067	517	483	283	70
FF	Mercaptobenzimidazole Zinc salt/Quinoline	3240	2760	633	537	340	113
EE	Mercaptobenzimidazole/Quinoline	3290	2297	600	557	387	120
Y	Mercaptobenzimidazole, MBI	3787	3033	553	550	387	127
GG	Mercaptotolylimidazole/Quinoline	3200	3033	703	433	363	133
NN	Mercaptotolylimidazole Zinc salt, ZMTI	3940	2847	533	517	350	77
VX	Mercaptotolylimidazole Zinc salt, ZMTI	3867	3373	783	540	473	123
MX	Mercaptotolylimidazole Zinc salt, ZMTI	3408	2958	621	537	405	90
AX	Mercaptotolylimidazole, MTI	3630	2977	547	540	387	120
AA	Mercaptotolylimidazole, MTI	3607	2317	497	540	383	120
Z	Mercaptobenzimidazole Zinc salt, ZMBI	3673	2703	387	517	333	97
XX2	miscellaneous	4183	2430	703	523	337	87
HH	miscellaneous	3537	2447	433	510	320	63
N	Mixed Ditolyl PPD	3750	1737	533	480	233	60
W	Naphthylamine	3910	2110	337	513	253	73
FX	Naphthylamine	3770	2733	598	490	317	77
R	Phenyl-a-Naphthylamine	3767	1170	457	480	217	60
OO	Quinoline	3977	2270	493	503	290	63
RR	Quinoline	3693	2287	505	487	313	83
D	Quinoline	3683	2540	558	457	283	67
CX	Quinoline	3655	2777	617	500	337	80
BB	Quinoline	3447	2390	503	507	297	83
HX	Quinoline/Diphenylamine	3810	2613	580	540	323	73
IX	Quinoline/Hindered Phenol	3933	2550	553	537	320	63
I	Quinoline/Dialkyl PPD	3867	2650	510	510	317	60
E	Quinoline/Diaryl PPD	3083	2300	467	447	290	50
F	Quinoline/Diaryl PPD/Dialkyl PPD	3733	3650	993	567	517	160
GX	Rx Prod of Diphenylamine, acetone & formaldehyde	3600	2280	637	510	347	77

Stress relaxation measurements are often used as a general guide to aging. The measurement basically consists of monitoring the stress in a sample while subjecting it to an accelerated aging procedure. Under suitable conditions, when viscous flow is not dominant, it has been proposed that the reactions within the rubber network may be related to stress changes as follows. The decay of stress in continuous relaxation measurements provides a measure of the degradative reactions in the network. If any new network is formed, it is considered to be in equilibrium with the main network and does not impose any new stress. Therefore, the decreasing stress level is a measure of the degree of chain scission, or degradation due to aging.

Stress relaxation experiments were conducted on most formulations at 250°F under a 50% strain using a Wallace Shawbury relaxometer. This instrument provides continuous reading of stress decay while keeping a constant strain on the specimen tested. The time to reach 30% and 70% relaxation of the original stress was determined and used to rank the oxidative stability of the antioxidants under static, strained conditions. Table 7 exhibits these results for 95 different antidegradants including blends of antioxidants and antiozonants. Analysis of the results shown in Table 7 indicate that some of the most effective antidegradant included:

- Substituted paraphenylenediamines, PPDs
- Blends of carbonyl amine condensate products
- Reaction product of acetone and diphenylamine
- Mercaptobenzimidazole/Mercaptotolylidimazole
- Mixture of Mercaptotolylimidazole with polymerized dihydroquinoline
- Mixture of Quinolines with Alkyl-Aryl PPDs.

In addition, a differential scanning calorimetry curve was developed for the most effective antioxidant systems using a Dupont 9900 Thermal Analysis System. Each material was tested using nitrogen as the purging gas from 40°C to 400°C. Table 8 depicts the heat of reaction and the peak temperatures of each different compound.

Table 7. Continuous Stress Relaxation at 50% Constant Strain

FORMULATION ID	COMMERCIAL NAME	ANTIOXIDANT TYPE	F/FO @250°F 70%	30%
JJ	Agerite Stalite	Diphenylamine	225	748
HH	Agerite SKT	miscellaneous	213	740
KK	Antozite 67	Alkyl-Aryl PPD	175	668
II	Agerite Stalite S	Diphenylamine	173	608
MM	Santoflex 13	Alkyl-Aryl PPD	165	598
B	Antozite2	Dialkyl PPD	155	633
J	Agerite Superflex Solid G/Antozite2	Carbonyl-amine Cond Prod/Dialkyl PPD	155	622
EE	Antioxidant MB/Agerite Resin D	Mercaptobenzimidaazole/Quinoline	148	705
F	Agerite Resin D/White/Antozite2/Agemaster	Quinoline/Diaryl PPD/Dialkyl PPD	147	593
LL	Agerite Resin D/Santoflex	Alkyl-Aryl PPD/Quinoline	145	718
FF	Antioxidant ZMB/Agerite Resin D	Mercaptobenzimidazole Zinc salt/Quinoline	145	675
H	Agerite Hipar/Antozite2	Blended Amine/Dialkyl PPD	145	578
Y	Antioxidant MB (Mobay)	Mercaptobenzimidazole, MBI	143	688
GG	Vanox MTI/Agerite Resin D	Mercaptotolylidimazole/Quinoline	142	668
W	Additin 30	Naphthylamine	142	573
AA	Vanox MTI	Mercaptotolylimidazole, MTI	140	705
EX	Naugard 477	Blended Amines	138	570
FX	Naugard PAN	Naphthylamine	138	563
JJX	Agerite Superflex Solid G/Resin D/Vanox MTI	Cond Prod/Quinoline/MTI	135	630
IX	Naugard BXA/Naugard Q	Quinoline/Hindered Phenol	133	523
Z	Antioxidant ZMB (Mobay)	Mercaptobenzimidazole Zinc salt, ZMBI	130	628
I	Santoflex AW/Antozite2	Quinoline/Dialkyl PPD	130	550
M	Wingstay 300/Antozite2	Alkyl-Aryl PPD/Dialkyl PPD	130	535
CC	Ethyl Antioxidant 703	Hindered Bisphenol	128	585
YY	Permanax IPPD	Alkyl-Aryl PPD	128	480
BB	Agerite MA	Quinoline	125	552
SS	Permanax BL	Carbonyl Amine Condensation Product	125	517
KX	Flexzone 7L	Alkyl-Aryl PPD	125	505
X	BLE 75	Carbonyl-amine Condensation Product	123	552
RR	Permanax TQ	Quinoline	123	513
BX	Vulcanox 4020	Alkyl-Aryl PPD	123	510
XX	Permanax DPPD	Diaryl PPD	123	500
QQ	Akrochem Antioxidant 36	Hindered Phenol	122	488
AX	Vulcanox MB-2/MG	Mercaptotolylimidazole, MTI	120	605
JX	Flexzone 12L	Blend of PPD	118	485
IIX	Agerite MA/Santoflex 13/Vanox MTI	Alkyl-Aryl PPD/Quinoline/MTI	117	683
PP	Akrochem Antioxidant S	Diphenylamine	115	497
V	Akrochem Antioxidant PD-2/Santoflex 13	Alkyl-Aryl PPD	113	490
OO	Akrochem Antioxidant DQ	Quinoline	112	472
L	Agerite White/Super Solid G/Antozite2	Cond Prod/Dialkyl PPD/Diaryl PPD	112	450
VV	Permanax WSL	Hindered Phenol	110	483
LX	Vulcanox 4030	Dialkyl PPD	110	480
DD	Ethyl Antioxidant 736	Hindered Bisphenol	108	465
CX	Naugard Q	Quinoline	108	453
ZZ	Vulcanox 4010 NA	Alkyl-Aryl PPD	106	438
HX	Naugard Q/Naugard 445	Quinoline/Diphenylamine	105	470
DX	Naugard 445	Diphenylamine	105	438
OOX	Santoflex 134D/Agerite Resin D/Vanox ZS	Alkyl-Aryl PPD/Quinoline/Hindered Phenol	102	528
WW	Permanax CNS	Blend of Non-staining Antioxidants	102	478
GX	Naugard BXA	Rx Prod of Diphenylamine, acetone & formaldehyde	100	452
K	Agerite Hipar T/Santoflex AW/Antozite2	Cond Prod/Quinoline/Dialkyl PPD	98	470
KKX	Agerite Stalite S/Resin D/Vanox MTI	Diphenylamine/Quinoline/MTI	95	405
TT	Permanax WSP	Hindered Bisphenol	93	428
LLX	Vanox 1320/Santoflex 13/Agerite Resin D	Alkyl-Aryl PPD/Quinoline/Hindered Bisphenol	92	520
NN	Akrochem Antioxidant 58	Mercaptotolylimidazole Zinc salt, ZMTI	90	563
QQX	Santoflex 134D/Agerite Resin D/Vanox MTI	Alkyl-Aryl PPD/Quinoline/MTI	90	503
Q	Akrochem Antioxidant PD-1	Alkyl-Aryl PPD	90	392
PPX	Santoflex 134D/Agerite Resin D/Santoflex DD	Alkyl-Aryl PPD/Quinoline/Quinoline	88	450

Table 7. Continuous Stress Relaxation at 50% Constant Strain—Continued

FORMULATION ID	COMMERCIAL NAME	ANTIOXIDANT TYPE	F/FO @250°F 70%	F/FO @250°F 30%
T	Flexamine G	Blended Amines	88	395
UU	Permanax WSO	Hindered Bisphenol	88	383
NNX	Santoflex 123D/Agerite Resin D/Vanox 1290	Alkyl-Aryl PPD/Quinoline/Hindered Bisphenol	83	410
E	Agerite Resin D/White	Quinoline/Diaryl PPD	83	402
PX	Flexzone 15L	Blend of PPD	83	355
VX	Akrochem Antioxidant 58	Mercaptotolylimidazole Zinc salt, ZMTI	82	390
S	Agerite DPPD	Diaryl PPD	82	368
MX	Vanox ZMTI	Mercaptotolylimidazole Zinc salt, ZMTI	80	383
MMX	Santoflex 134D/Agerite Resin D/Vanox 102	Alkyl-Aryl PPD/Quinoline/Hindered Phenol	80	380
N	Wingstay 100	Mixed Ditolyl PPD	80	378
XX2	Ethanox 330	miscellaneous	78	345
CCX	Antozite 67	Alkyl-Aryl PPD	77	347
WX	Aminox 5664	Condensation Products	75	330
RX	Flexzone 3C	Alkyl-Aryl PPD	75	318
HHX	Agerite Resin D/Santoflex 13/Vanox MTI	Alkyl-Aryl PPD/Quinoline/MTI	72	310
G	Agemaster		72	298
C	Agerite White	Diaryl PPD	68	312
U	Agerite HPS	Blended Amines	67	320
D	Agerite Resin D	Quinoline	65	403
P	Akrochem Antioxidant 2246	Hindered Bisphenol	65	320
R	Akrochem Antioxidant PANA	Phenyl-a-Naphthylamine	63	285
SSX	Santoflex 13/Agerite Resin D/DTDTDP	Alkyl-Aryl PPD/Quinoline	62	353
RRX	Santoflex 134D/Agerite Resin D/Santoflex 13	Alkyl-Aryl PPD/Quinoline/Alkyl-Aryl PPD	60	275
ZX	Ethanox 702	Hindered Bisphenol	58	252
OX	Permanax BL	Carbonyl-amine Condensation Product	58	215
O	Octamine	Diphenylamine	57	310
DDX	BLE 25/Flexamine G	Carbonyl-amine Cond Prod/Blended Amine	55	230
BBX	Ethanox 701	Hindered Phenol	52	245
AAX	Ethanox 744		47	223
TX	Flexzone 11L	Blend of PPD	45	190
A	None		40	238
QX	Flexzone 6H	Alkyl-Aryl PPD	40	180
UX	Flexzone HS		40	180
FFX	Santoflex 13/Agerite Resin D	Alkyl-Aryl PPD/Quinoline	33	168
GGX	Santoflex 13/Agerite MA	Alkyl-Aryl PPD/Quinoline	33	138
SX	Flexzone 4L	Dialkyl PPD	33	115
EEX	Santoflex 13	Alkyl-Aryl PPD	30	140

Table 8. Differential Scanning Calorimetry Data

FORMULATION ID	COMMERCIAL NAME	PEAK TEMPERATURE (C)	JOULES/G	MINOR EVENT (C)	MINOR EVENT JOULES/G
LL	Agerite Resin D/Santoflex	386.68	42.01	210.74	3.10
LLX	Vanox 1320/Santoflex 13/Agerite Resin D	394.45	53.16	219.01	0.338
JJX	Agerite Superflex Solid G/Resin D/Vanox MTI	388.6	55.3		
EEX	Santoflex 13	387.83	56.32	211.66	3.635
RRX	Santoflex 134D/Agerite Resin D/Santoflex 13	391.57	56.6	213.36	3.425
HHX	Agerite Resin D/Santoflex 13/Vanox MTI	396.46	63.39	212.76	4.011
DDX	BLE 25/Flexamine G	392.62	68.93	211.66	2.572
KKX	Agerite Stalite S/Resin D/Vanox MTI	388.65	69.61	208.65	3.317
K	Agerite Hipar T/Santoflex AW/Antozite2	289.44	69.74		
OOX	Santoflex 134D/Agerite Resin D/Vanox ZS	390.75	74.81	160.57	10.18
GGX	Santoflex 13/Agerite MA	393.81	75.13		
QQX	Santoflex 134D/Agerite Resin D/Vanox MTI	389.98	75.26	159.71	2.594
MMX	Santoflex 134D/Agerite Resin D/Vanox 102	381.46	75.97		
V	Akrochem Antioxidant PD-2/Santoflex 13	388.44	76.01	212.76	4.296
EE	Antioxidant MB/Agerite Resin D	390.41	76.91	210.54	3.711
PPX	Santoflex 134D/Agerite Resin D/Santoflex DD	389.27	81.16	205.58	7.763
IIX	Agerite MA/Santoflex 13/Vanox MTI	391.22	82.46	210.8	1.74
SSX	Santoflex 13/Agerte Resin D/DTDTDP	391.17	83.58	210.74	2.204
F	Agerite Resin D/White/Antozite2/Agemaster	390.8	83.85		
GG	Vanox MTI/Agerite Resin D	388.02	95.9	209.26	4.154
FF	Antioxidant ZMB/Agerite Resin D	388.03	96.87	211.9	3.848
A	None	388.2	103.3	163.32	4.519
NNX	Santoflex 134D/Agerite Resin D/Vanox 1290	384.08	121.5		
H	Agrrite Hipar/Antozite2	389.38	132.5	212.56	1.324

DISCUSSION OF RESULTS

An important finding among the group of antidegradants used was the apparent synergistic effect exhibited by MTI and a diphenylamine in formulation KKX. The tensile strength retention for this compound at 250°F was 27.7% (as shown in Table 3) which was far superior than any one of the antioxidants in the mixture by itself. Compound KKX contained a blend of Agerite Stalite S, Agerite Resin D, and Vanox MTI. These individual compounds produced lower tensile retention values than when combined. Compound II represents Agerite Stalite S by itself which produced a 13.6% retention, Compound D represents Agerite Resin D by itself which produced a 15.2% tensile strength retention, and Compound AA represents Vanox MTI by itself which produced a tensile strength of 13.8%. The synergism was even more pronounced with the mixture of PPD and TMQ as exhibited in Formulation LL. Compound LL produced a tensile retention at 250°F of 28%. Even though Santoflex AW was not tested alone, the combination produced a signficant improvement over Agerite Resin D by itself which produced only 15.2% retention. On the other hand, only one mixture of antioxidants behave in an antagonistic way. Compound V exhibited one of the lowest tensile strength retention at 250°F, only 8.2%, when compared with compound EEX containing Santoflex 13 which exhibited a retention of 17.3%. Table 4 presents the data for all compounds in decreasing order of tensile strength retention at 250°F.

Figures A-1 through A-11 (see Appendix) show the tensile strength data for different groups of antioxidants and mixtures. It is evident that the quinolines (TMQ and ZMTI) exhibit the best tensile retention after aging, while the naphthylamines produced the lowest retention after aging at 250°F. Figures A-12 through A-22 exhibit the ultimate elongation for the antioxidants, while Figures A-23 through A-33 show the product of tensile and elongation at 250°F. Nonetheless, the best retention of properties was achieved by mixing two or more antioxidants in a given formulation. This is demonstrated in Table 9 which presents the data for all the mixtures. It is important to note that some of the compounds that exhibit high tensile strength retention after aging at 250°F produced low original tensile strength. This is likely to be due to further cross-linking upon aging. Compounds with tensile retention in the order of 30%, 40%, and 50% produced the lowest original tensile strength, at a level not deemed adequate for tank pad applications. On the other hand, the group of compounds with tensile retention after aging at 250°F between 20-30% produced a good balance of properties. Significant among this group are compounds F, KKX, LL, RRX—all of which contain a mixture of a quinoline and a substituted para-phenylenediamine.

Table 8 depicts the data obtained from the differential scanning calorimetry test, DSC. The peak temperature and the heat of reaction related to the oxidative cross-linking and the oxidative degradation reactions are listed in decreasing order. The thermograms for the compounds listed in Table 8 are illustrated in Figures A-34 through A-57. In most of the compounds, two exotherms are evident in a nitrogen inert atmosphere. The initial exotherm, which occurs in the 180-200°C region

Table 9. Antioxidant Combinations

FORMULATION ID	COMMERICAL NAME	TENSILE STRENGTH ORIG (PSI)	@250°F (PSI)	ULTIMATE ELONG ORIG (%)	250°F (%)	RETENTION TENSILE (%)	ELONGATION (%)
QQX	Santoflex 134D/Agerite Resin D/Vanox MTI	1567	838	417	140	53.5	33.6
OOX	Santoflex 134D/Agerite Resin D/Vanox ZS	1850	773	417	100	41.8	24.0
IIX	Agerite MA/Santoflex 13/Vanox MTI	1983	803	503	130	40.5	25.8
PPX	Santoflex 134D/Agerite Resin D/Santoflex DD	2000	733	470	107	36.7	22.8
JJX	Agerite Superflex Solid G/Resin D/Vanox MTI	2217	790	480	123	35.6	25.6
MMX	Santoflex 134D/Agerite Resin D/Vanox 102	2200	767	460	97	34.9	21.1
NNX	Santoflex 134D/Agerite Resin D/Vanox 1290	2283	765	470	97	33.5	20.6
LLX	Vanox 1320/Santoflex 13/Agerite Resin D	2383	794	487	113	33.3	23.2
GGX	Santoflex 13/Agerite MA	2667	757	533	103	28.4	19.3
LL	Agerite Resin D/Santoflex	3327	933	583	143	28.0	24.5
KKX	Agerite Stalite S/Resin D/Vanox MTI	3683	1018	597	167	27.6	28.0
F	Agerite Resin D/White/Antozite2/Agemaster	3733	993	567	160	26.6	28.2
GG	Vanox MTI/Agerite Resin D	3200	703	433	133	22.0	30.7
DDX	BLE 25/Flexamine G	3633	775	560	98	21.3	17.5
RRX	Santoflex 134D/Agerite Resin D/Santoflex 13	3967	813	510	97	20.5	19.0
FF	Antioxidant ZMB/Agerite Resin D	3240	633	537	113	19.5	21.0
SSX	Santoflex 13/Agerite Resin D/DTDTDP	4183	787	523	97	18.8	18.5
EE	Antioxidant MB/Agerite Resin D	3290	600	557	120	18.2	21.5
HHX	Agerite Resin D/Santoflex 13/Vanox MTI	4183	753	573	90	18.0	15.7
H	Agerite Hiper/Antozite2	3700	637	493	87	17.2	17.6
K	Agerite Hiper T/Santoflex AW/Antozite2	3575	590	500	87	16.5	17.4
L	Agerite White/Super Solid G/Antozite2	3767	620	510	83	16.5	16.3
FFX	Santoflex 13/Agerite Resin D	3950	650	543	90	16.5	16.6
HX	Naugard Q/Naugard 445	3810	580	540	73	15.2	13.5
J	Agerite Superflex Solid G/Antozite2	3625	550	510	83	15.2	16.3
E	Agerite Resin D/White	3083	467	447	50	15.1	11.2
AX	Vulcanox MB-2/MG	3630	547	540	120	15.1	22.2
M	Wingstay 300/Antozite2	3733	540	513	67	14.5	13.1
IX	Naugard BXA/Naugard Q	3933	553	537	63	14.1	11.7
I	Santoflex AW/Antozite2	3867	510	510	60	13.2	11.8
V	Akrochem Antioxidant PD-2/Santoflex 13	3687	303	510	73	8.2	14.3

relates to the multiplicity of vulcanization reactions. The second exotherm which occurs in the 350-450°C region relates to the polymer degradation or chain scission. Compound LL depicted in Figure A-34 shows the lowest degradation exotherm from the compounds with antioxidant mixtures. Compound LL was also the compound that produced the best synergistic effect of antioxidants. Compound QQX, depicted in Figure A-45, with a mixture of three antioxidants was the only one producing a melting endotherm.

SECTION III. CONCLUSIONS

Very little correlation was found between the different methods used to rank the antioxidant efficiency. Thermal analysis provided information on thermal degradation in an inert atmosphere with temperatures high enough to activate cross-linking reaction. Oven aging was the more common of all tests conducted, but careful interpretation of the results is necessary since tensile strength and elongation retention did not always point in the same direction. On the other hand, stress relaxation provided an aging environment with the specimens under constant strain, a more realistic scenario. Nevertheless, regardless of the method of evaluation, the substituted phenylene diamines and the quinolines were the antioxidant groups with the best performance. In many cases, the combination of one or more of these compounds produced excellent results. The substituted paraphenylene diamines protected the rubber vulcanizates against ozone and oxygen, as well as fatigue, heat, and metal ions. Santoflex 134D was outstanding among the group, closely followed by Agerite White.

To achieve the maximum high temperature protection for tank pad vulcanizates, the optimization of a given antioxidant system is necessary. Some of the best candidates of antioxidants include:

1. Agerite [Stalite, Resin D, Superflex Solid G, MA, White and Hipar T].

2. Antozite 2 and Antozite 67

3. Santoflex 13 and Santoflex 134D

4. Vanox [MTI, ZS, 102, 1290. 1320]

5. BLE 25

6. Flexamine G

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APPENDIX OF FIGURES

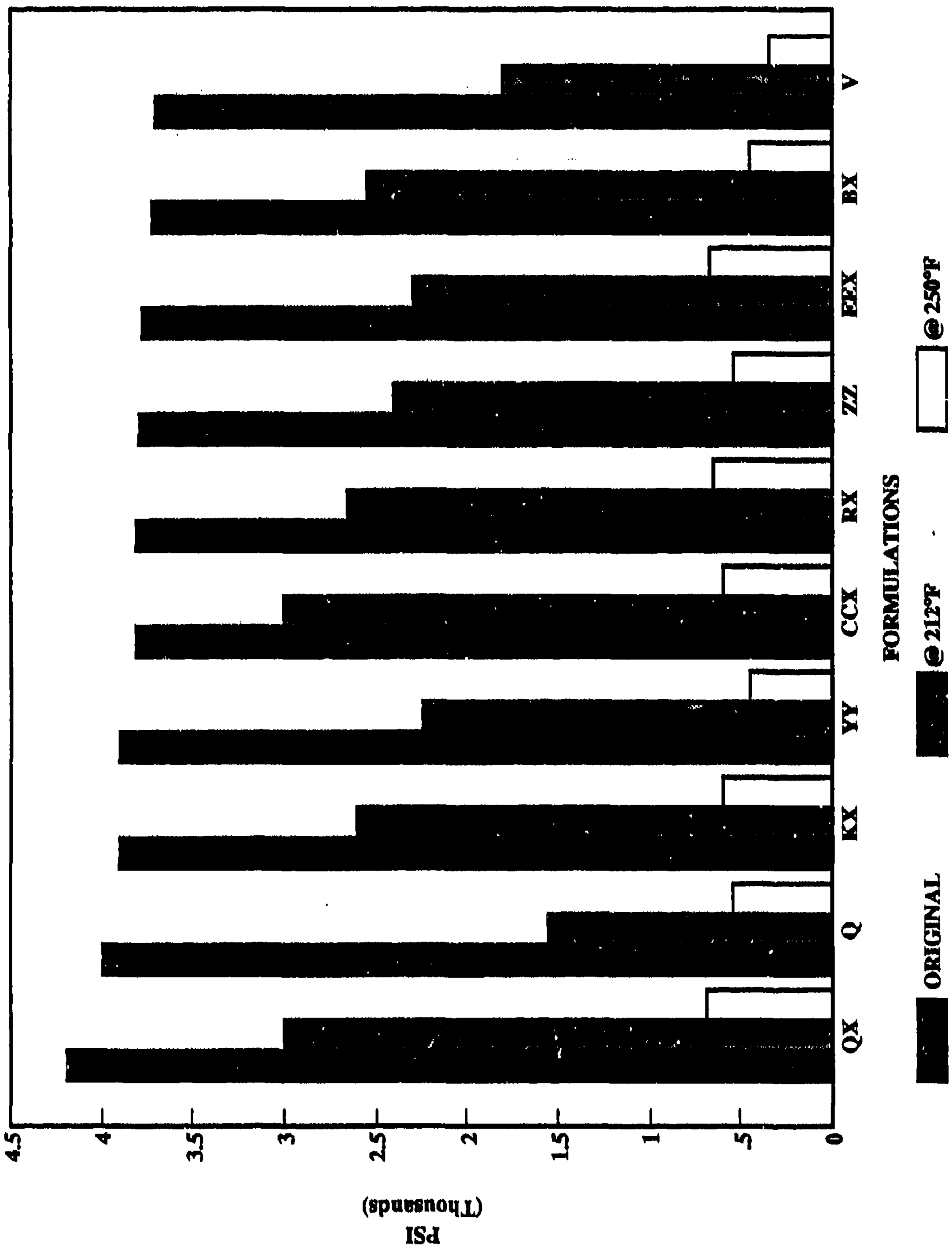


Figure A-1. Tensile Strength, Alkyl-Aryl PPDs

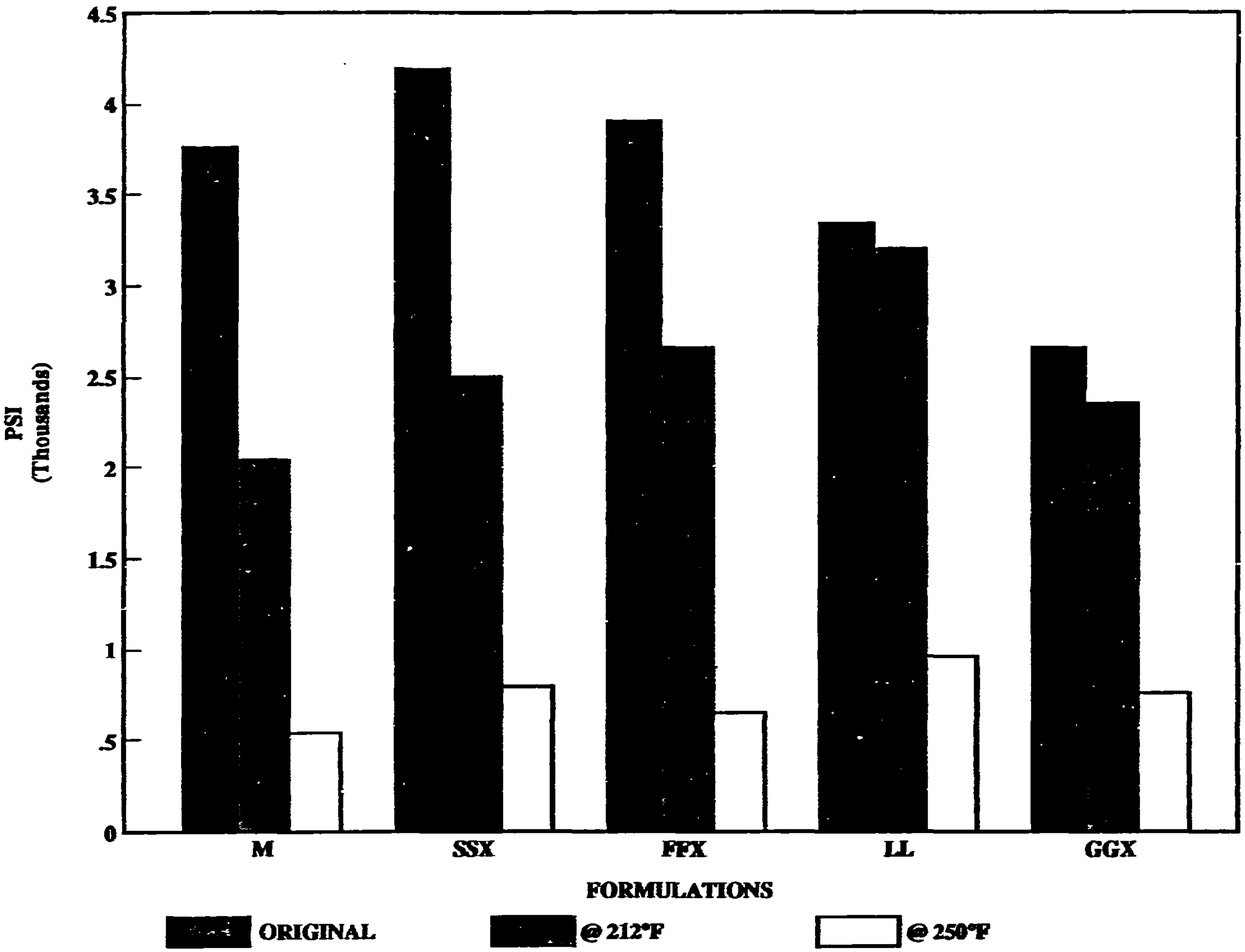


Figure A-2. Tensile Strength, Alkyl-Aryl PPDs/Quinolines

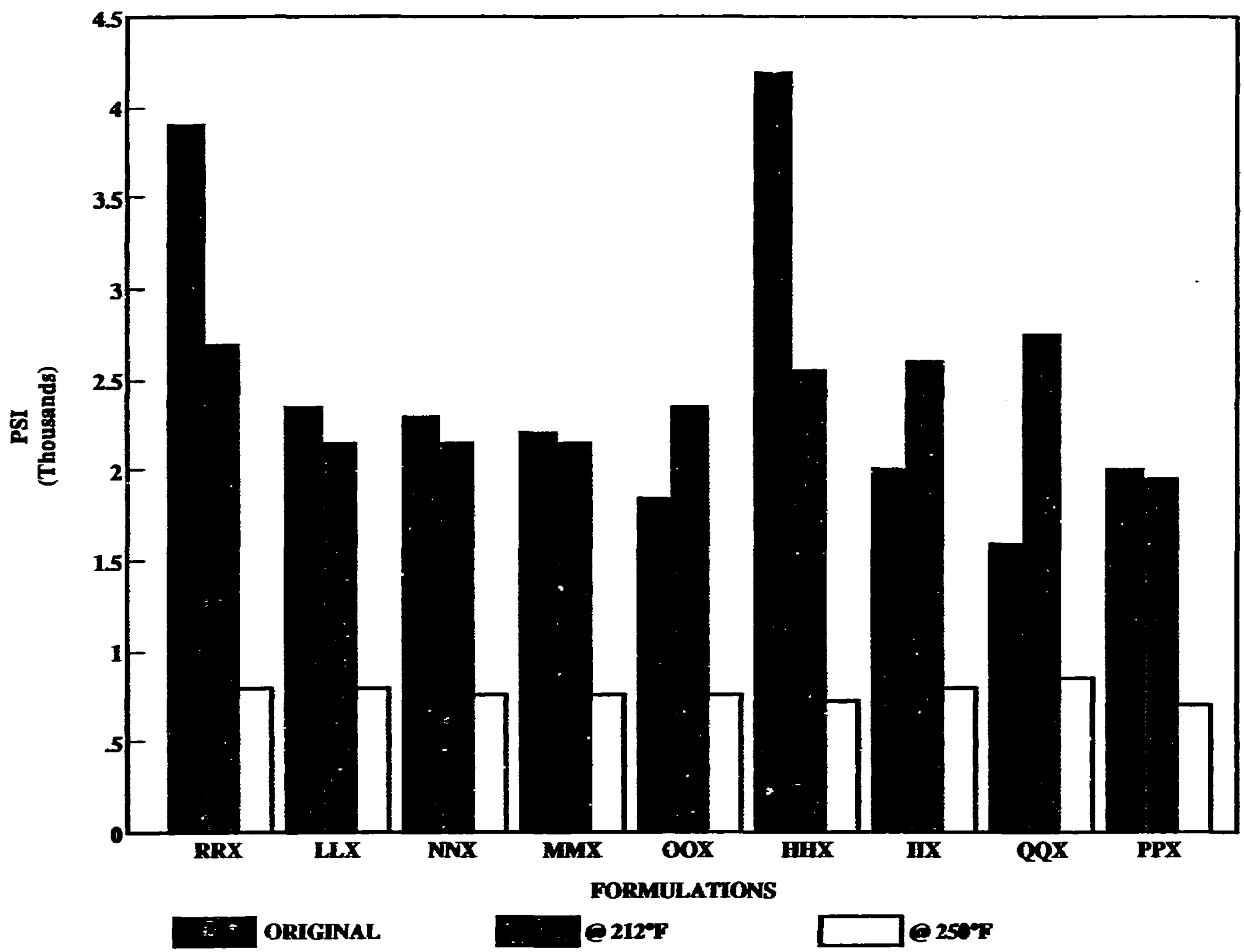


Figure A-3. Tensile Strength, Alkyl-Aryl PPDs/Quinolines/Others

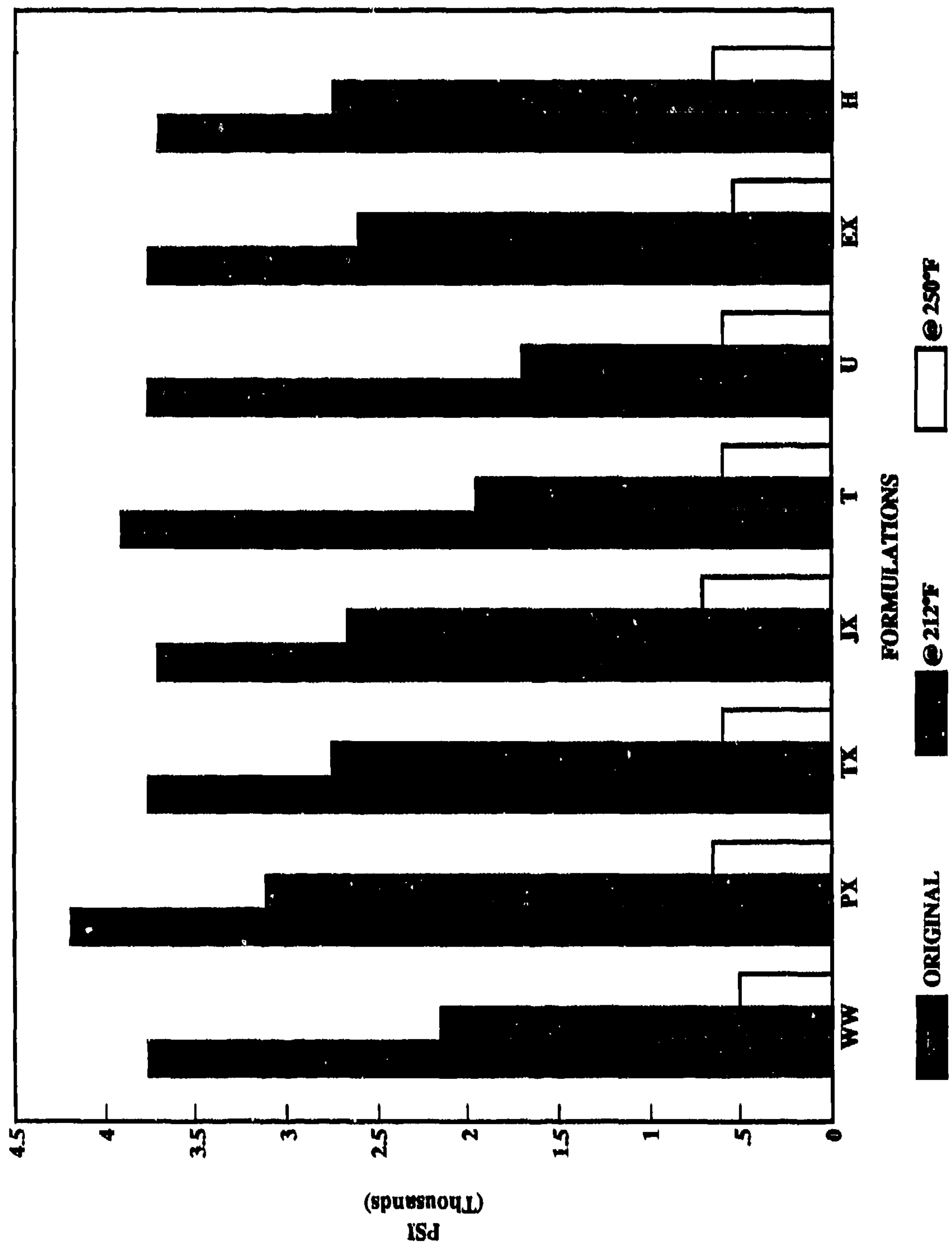


Figure A-4. Tensile Strength, Blended Amines

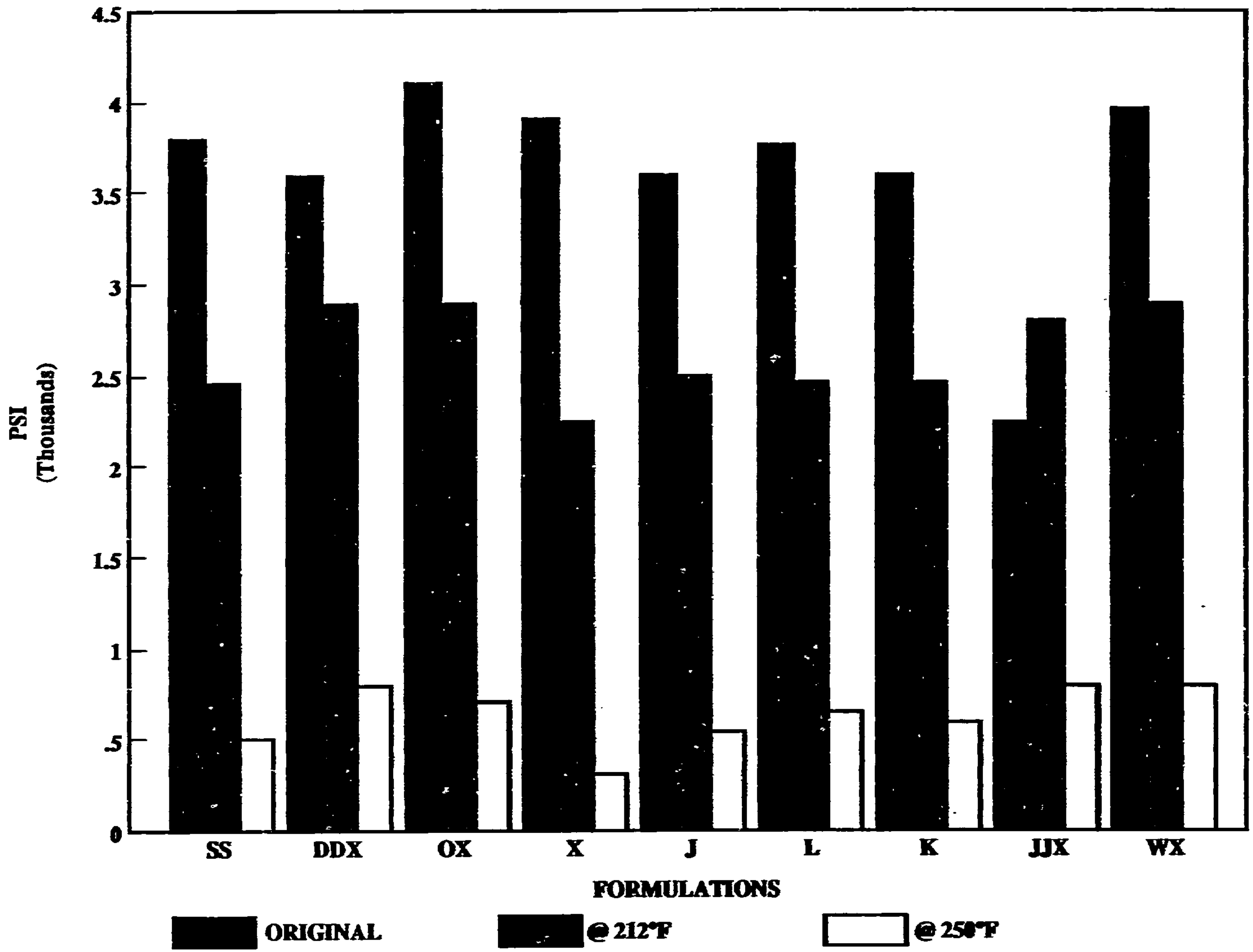


Figure A-5. Tensile Strength, Condensation Products/Others

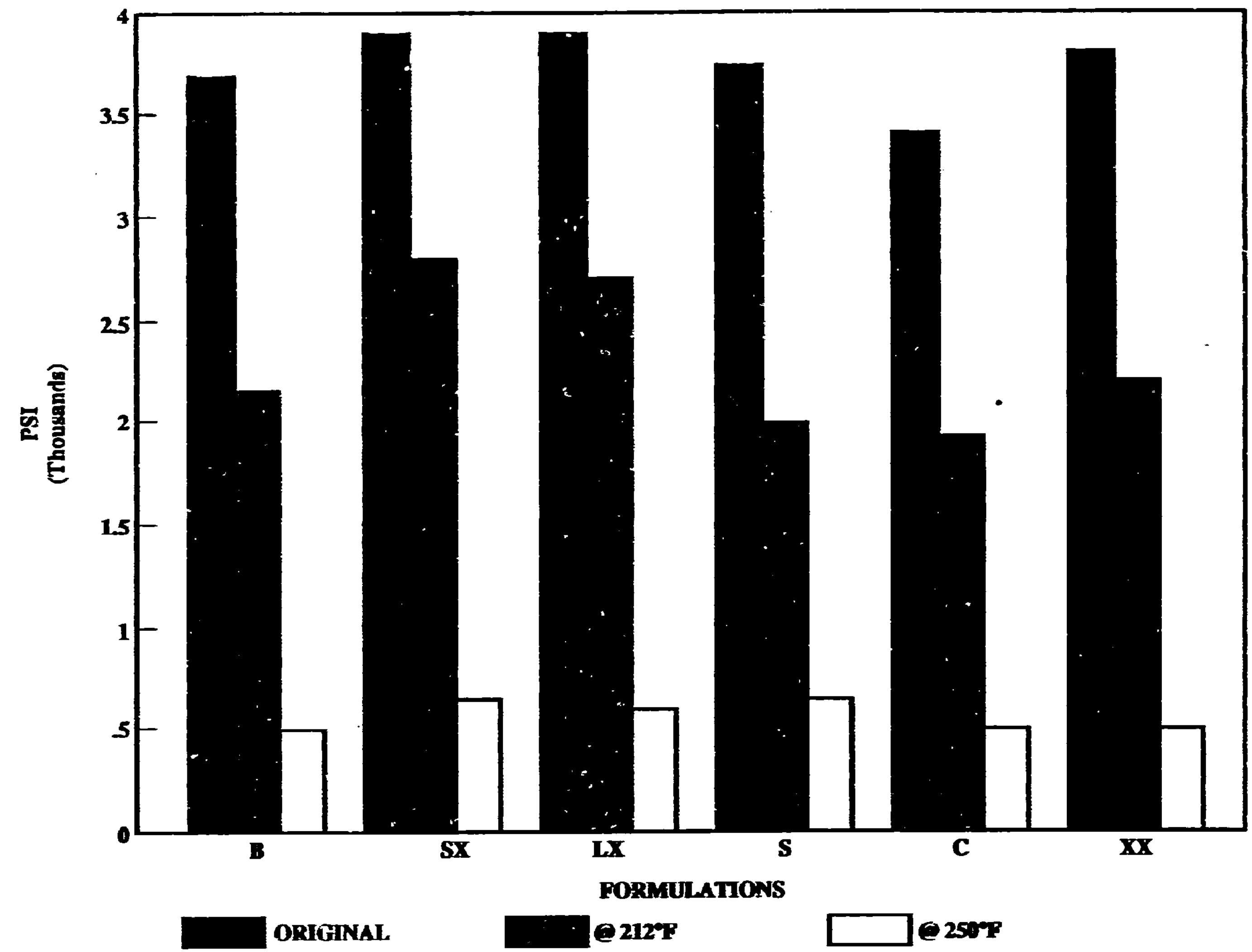


Figure A-6. Tensile Strength, Dialkyl and Diaryl PPDs

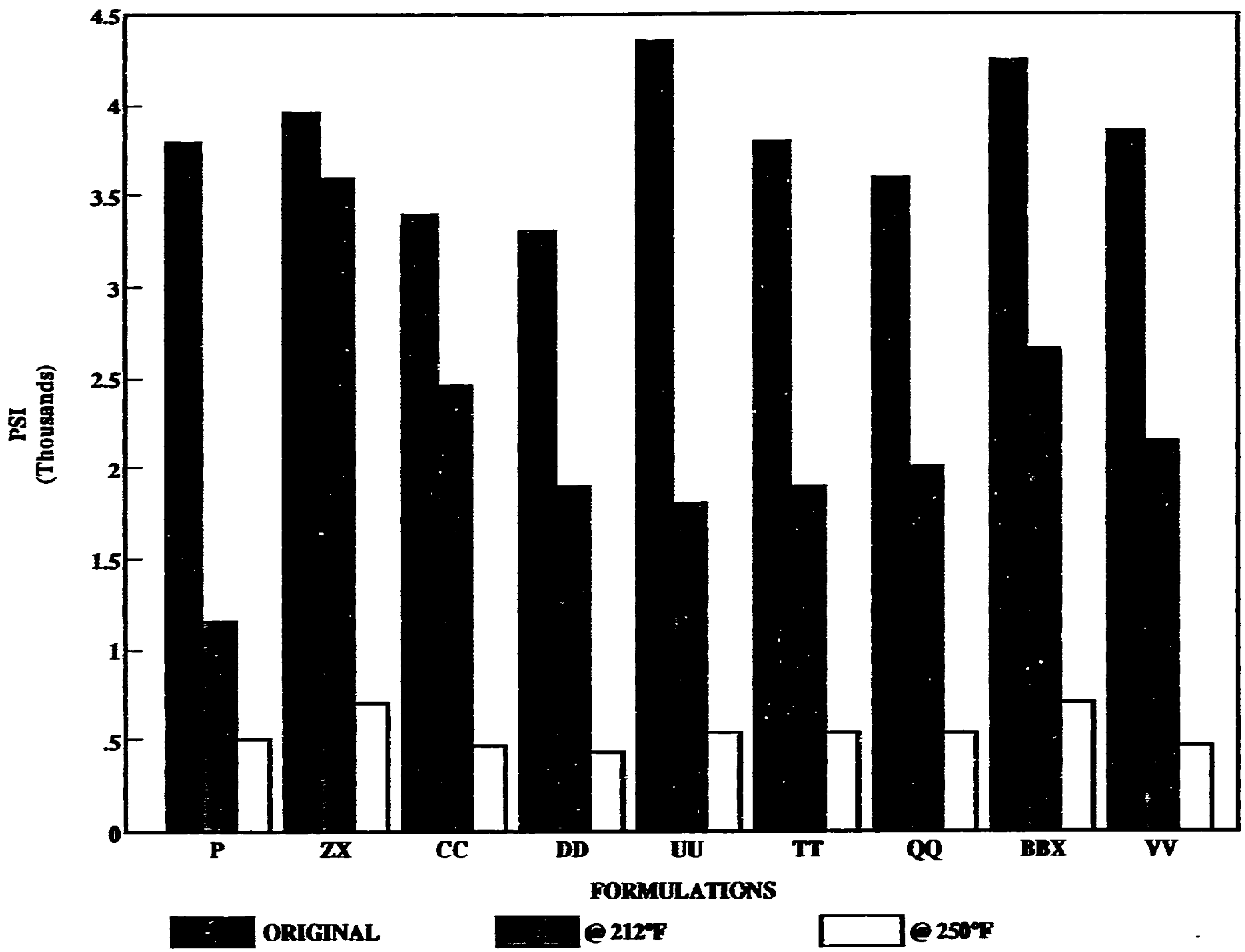


Figure A-7. Tensile Strength, Hindered Bisphenols and Phenols

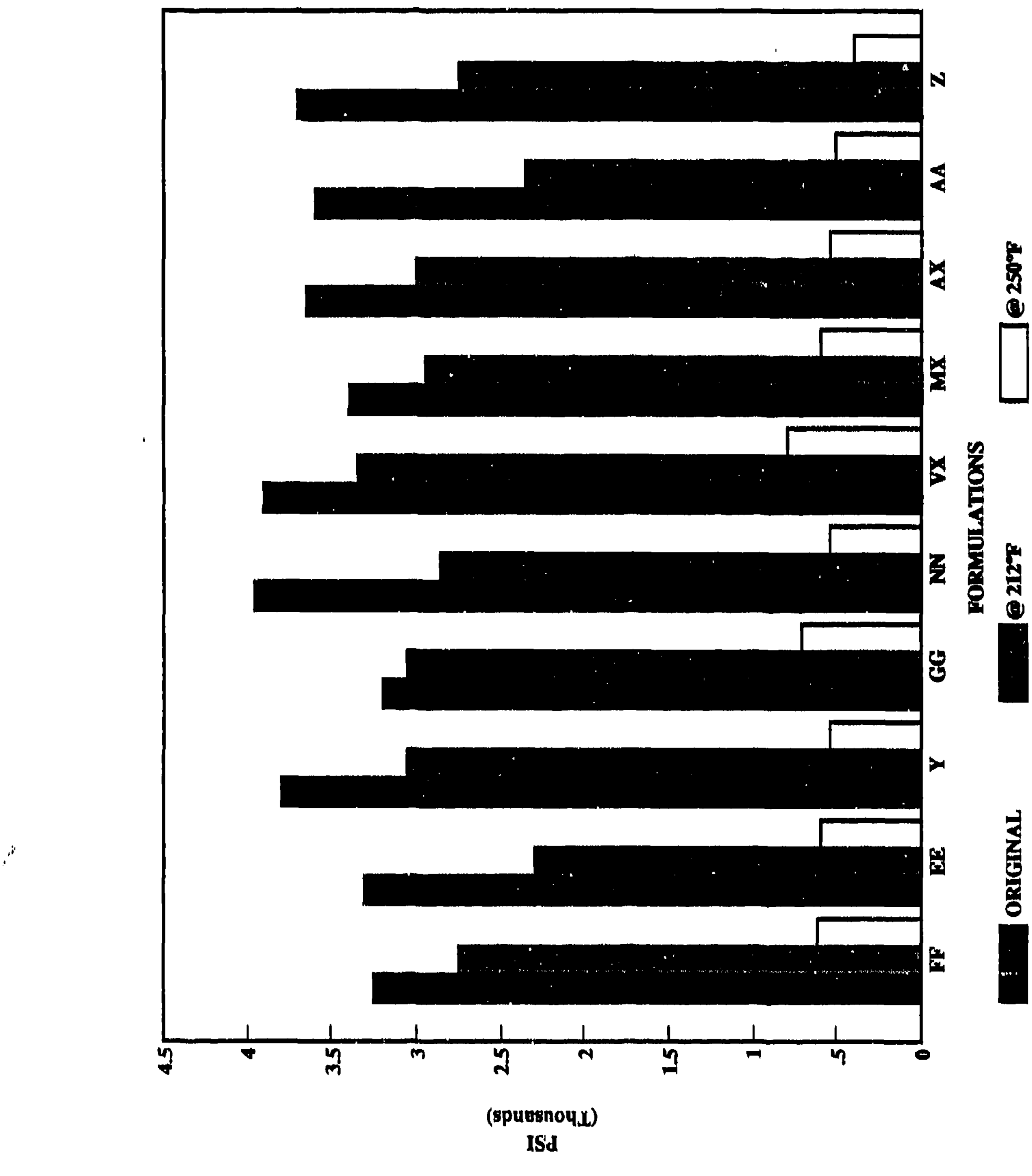


Figure A-8. Tensile Strength, MBI, ZMBI, MTI, ZMTI

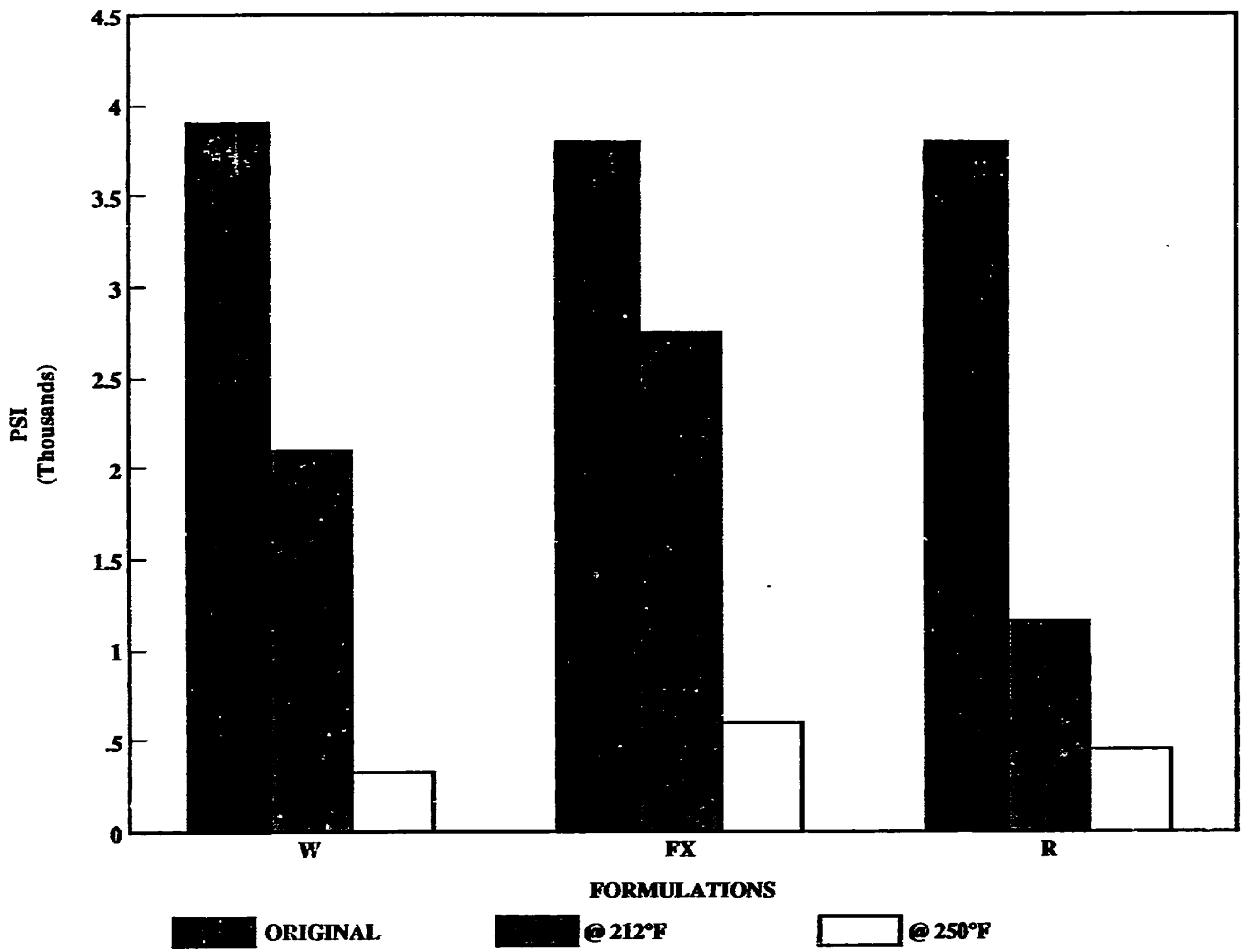


Figure A-9. Tensile Strength, Naphthylamines

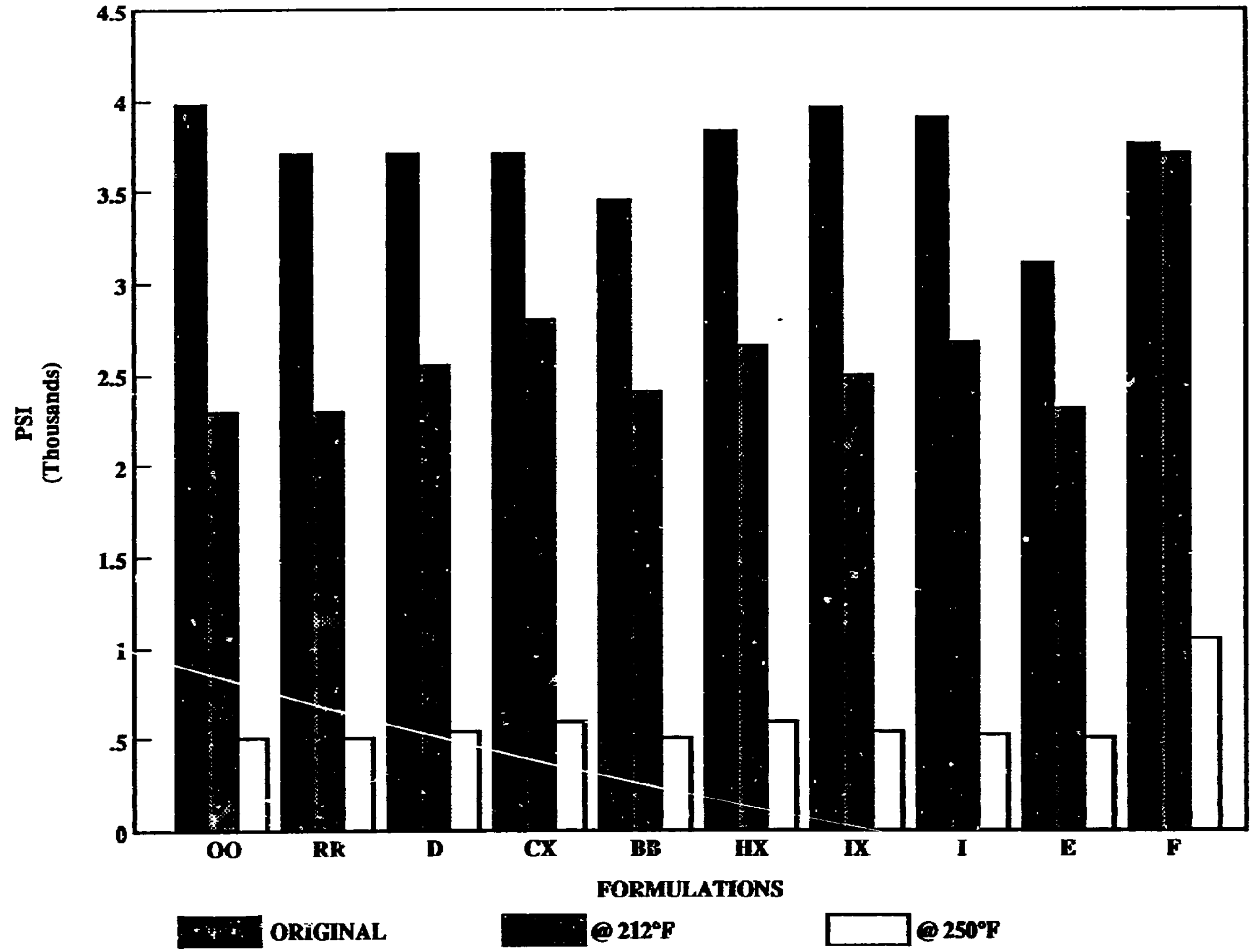


Figure A-10. Tensile Strength, Quinolines and Blends

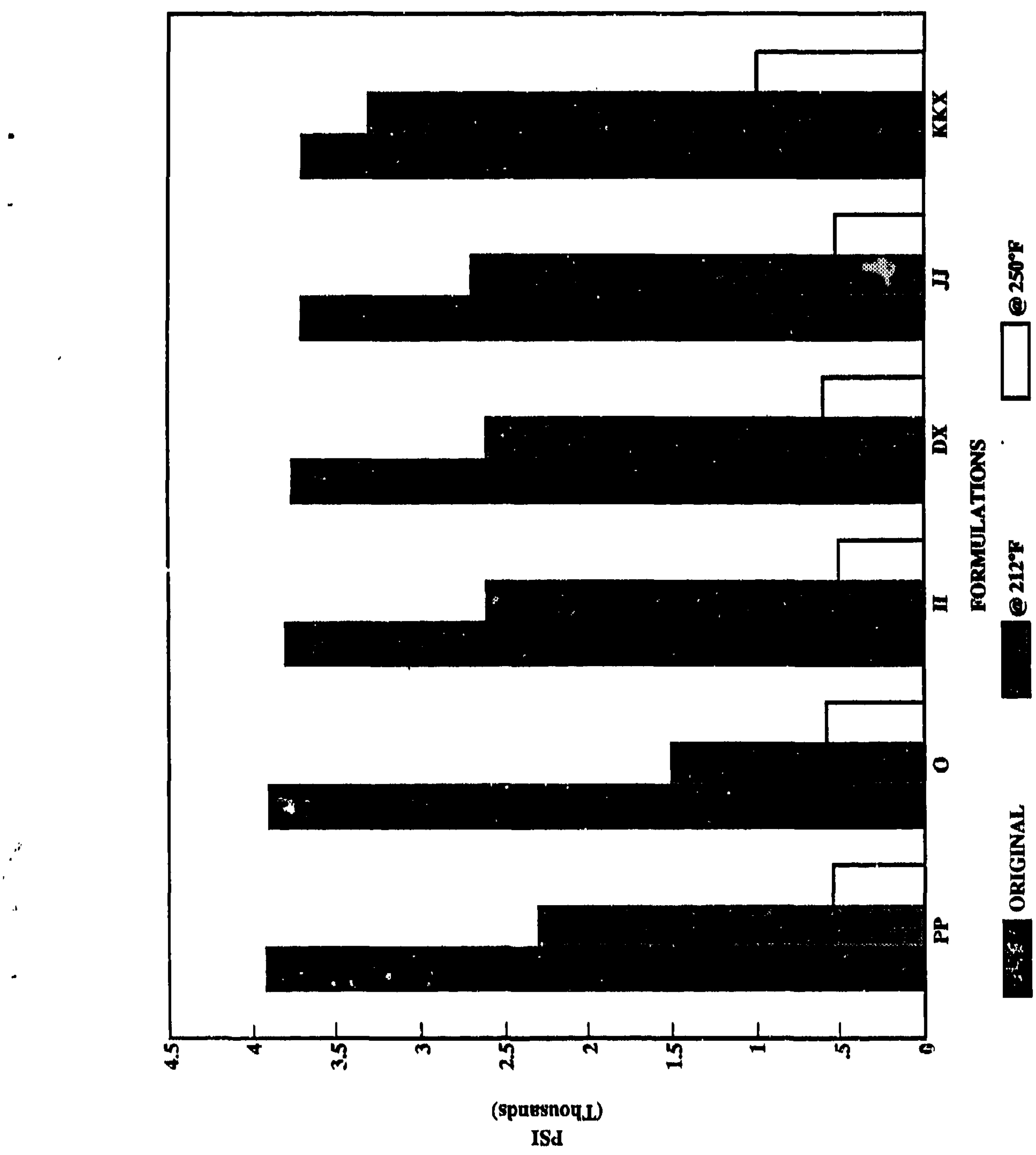


Figure A-11. Tensile Strength, Diphenylamines

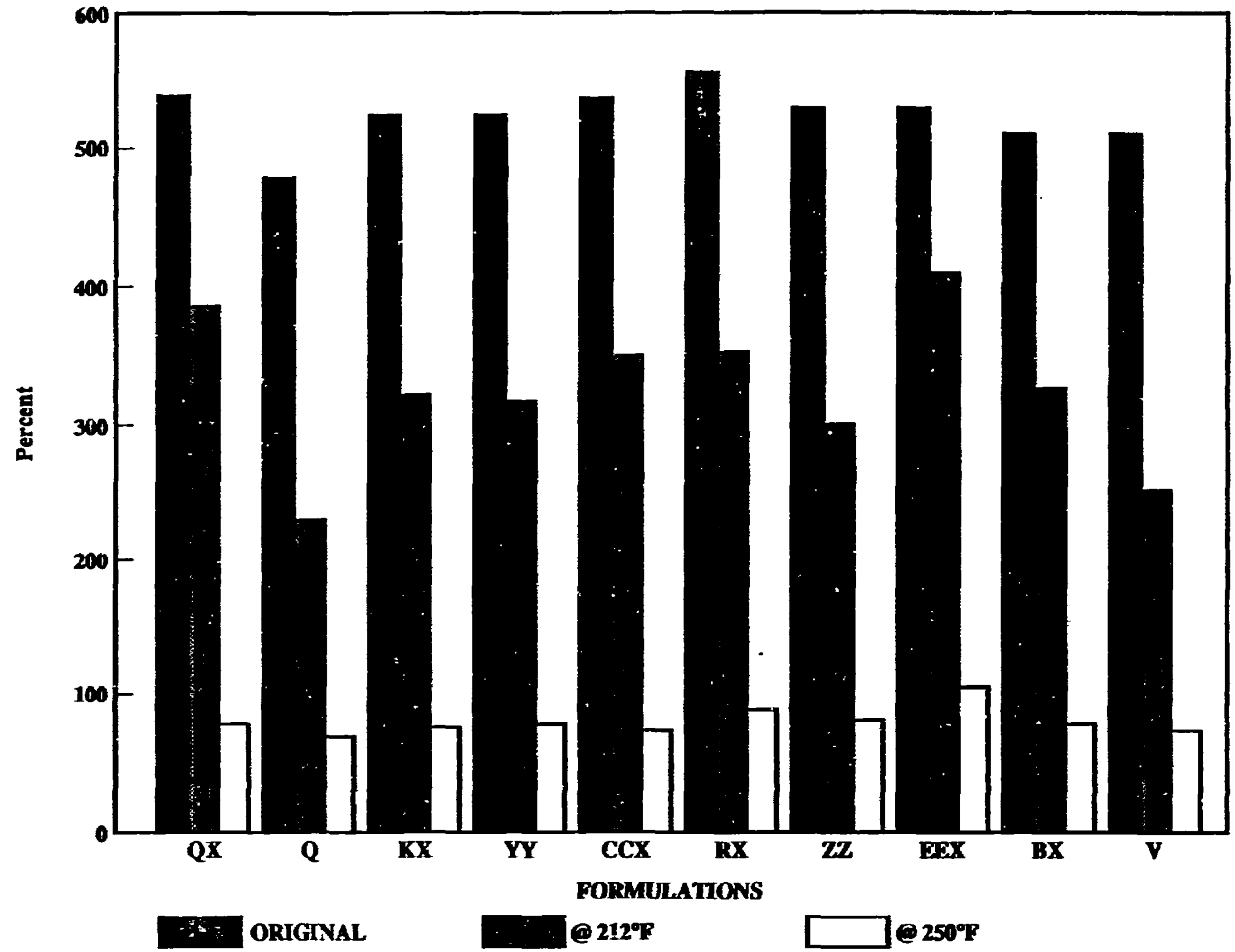


Figure A-12. Ultimate Elongation, Alkyl-Aryl PPDs

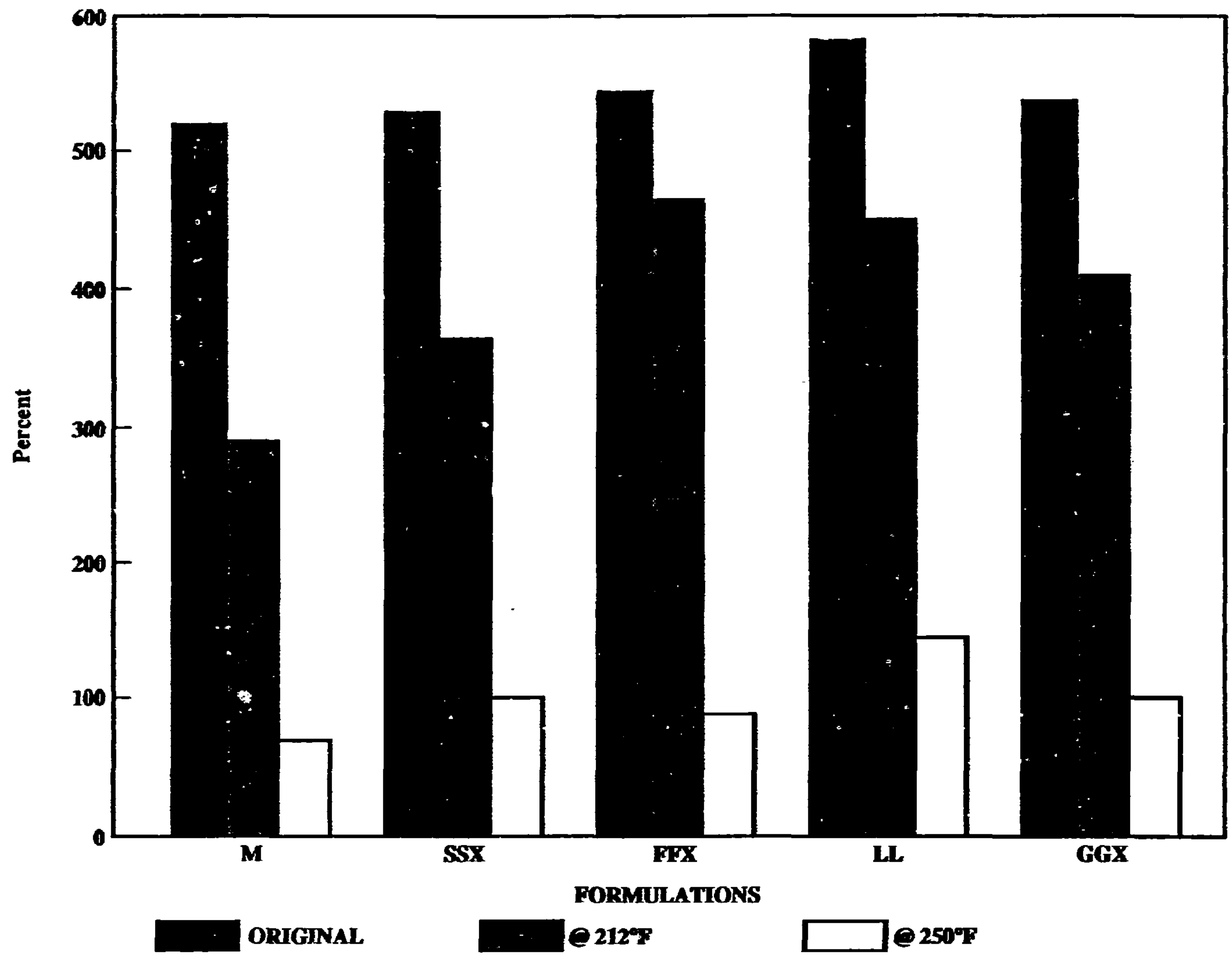


Figure A-13. Ultimate Elongation, Alkyl-Aryl PPDs/Quinolines

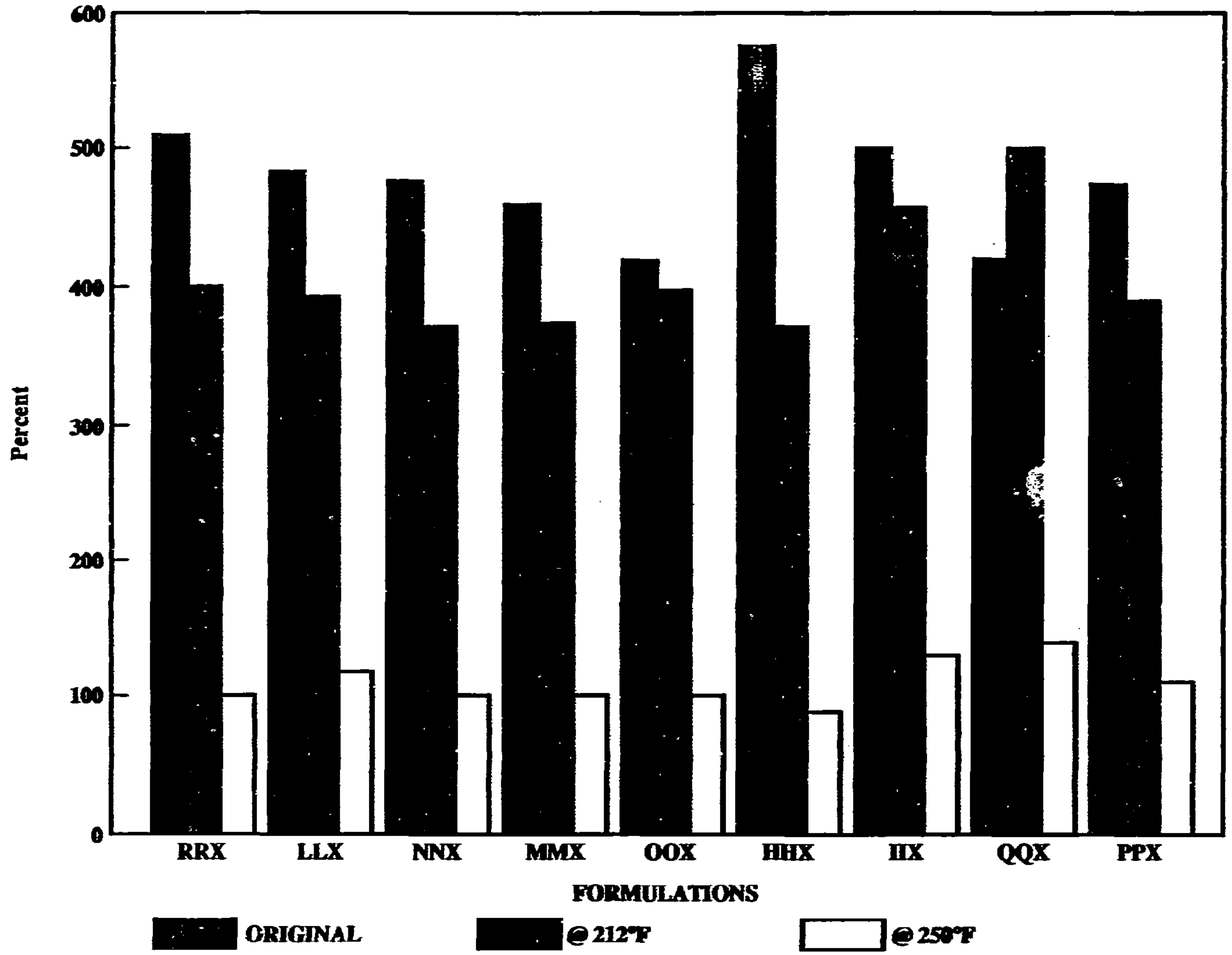


Figure A-14. Ultimate Elongation, Alkyl-Aryl PPDs/Quinolines/Others

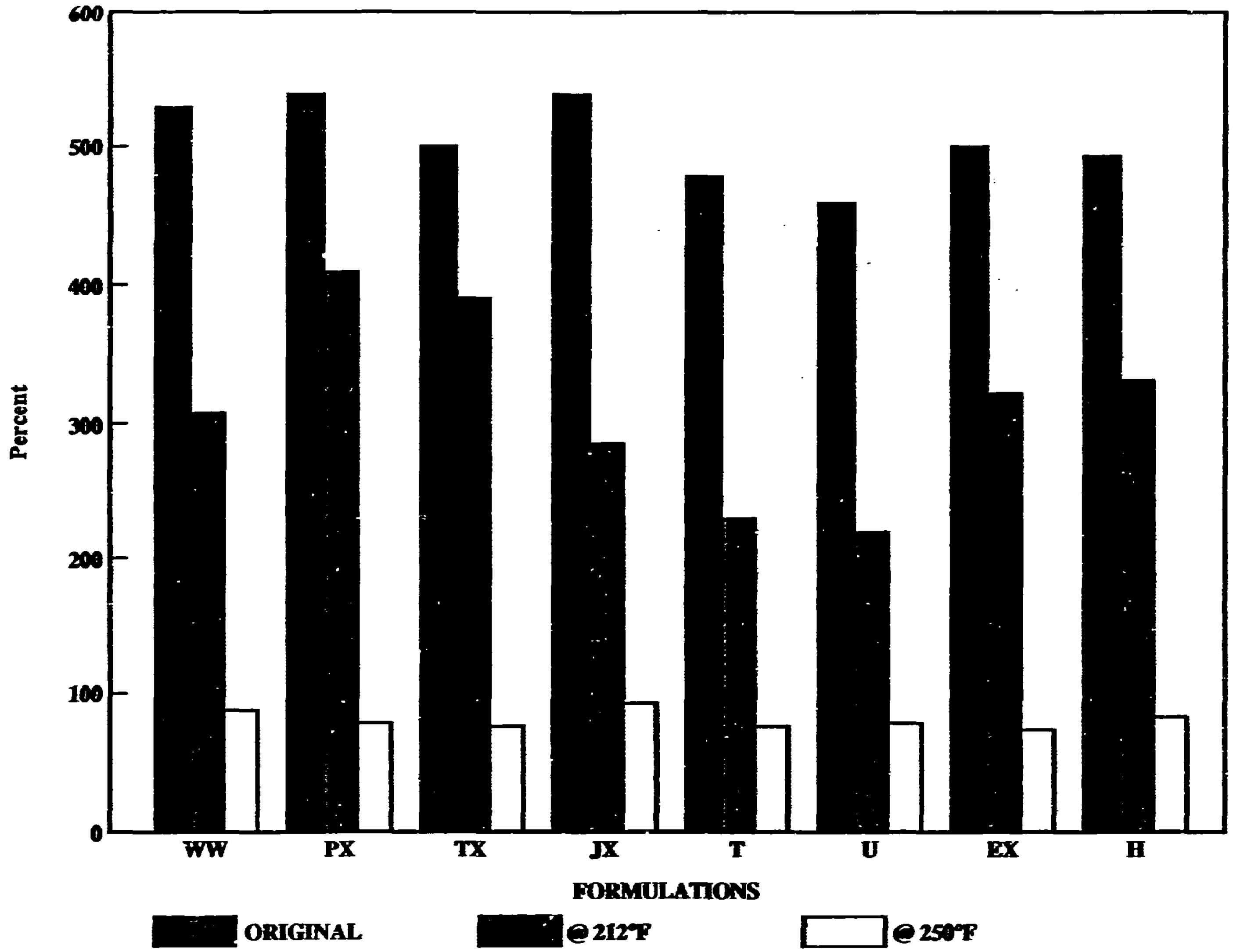


Figure A-15. Ultimate Elongation, Blended Amines

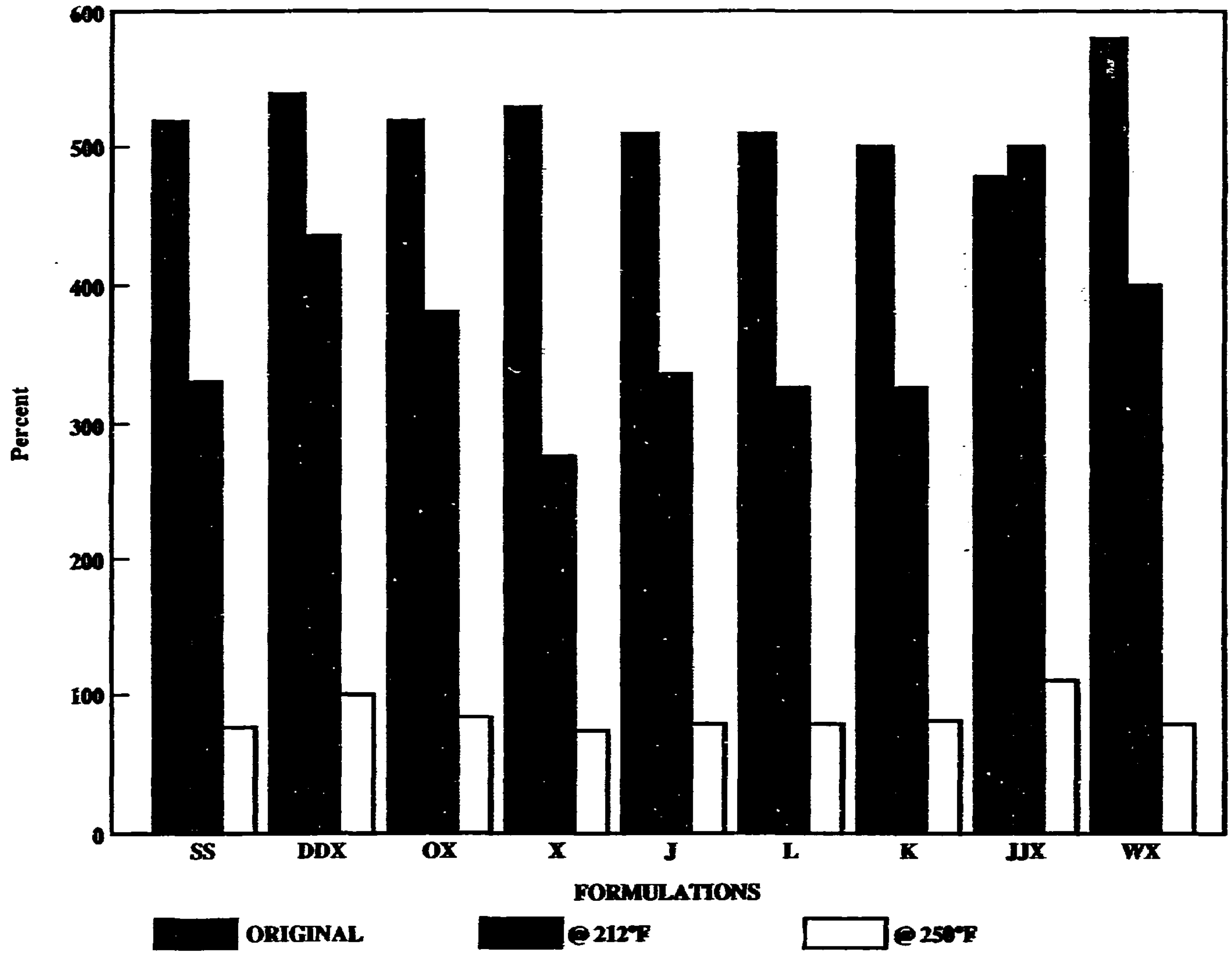


Figure A-16. Ultimate Elongation, Condensation Products/Others

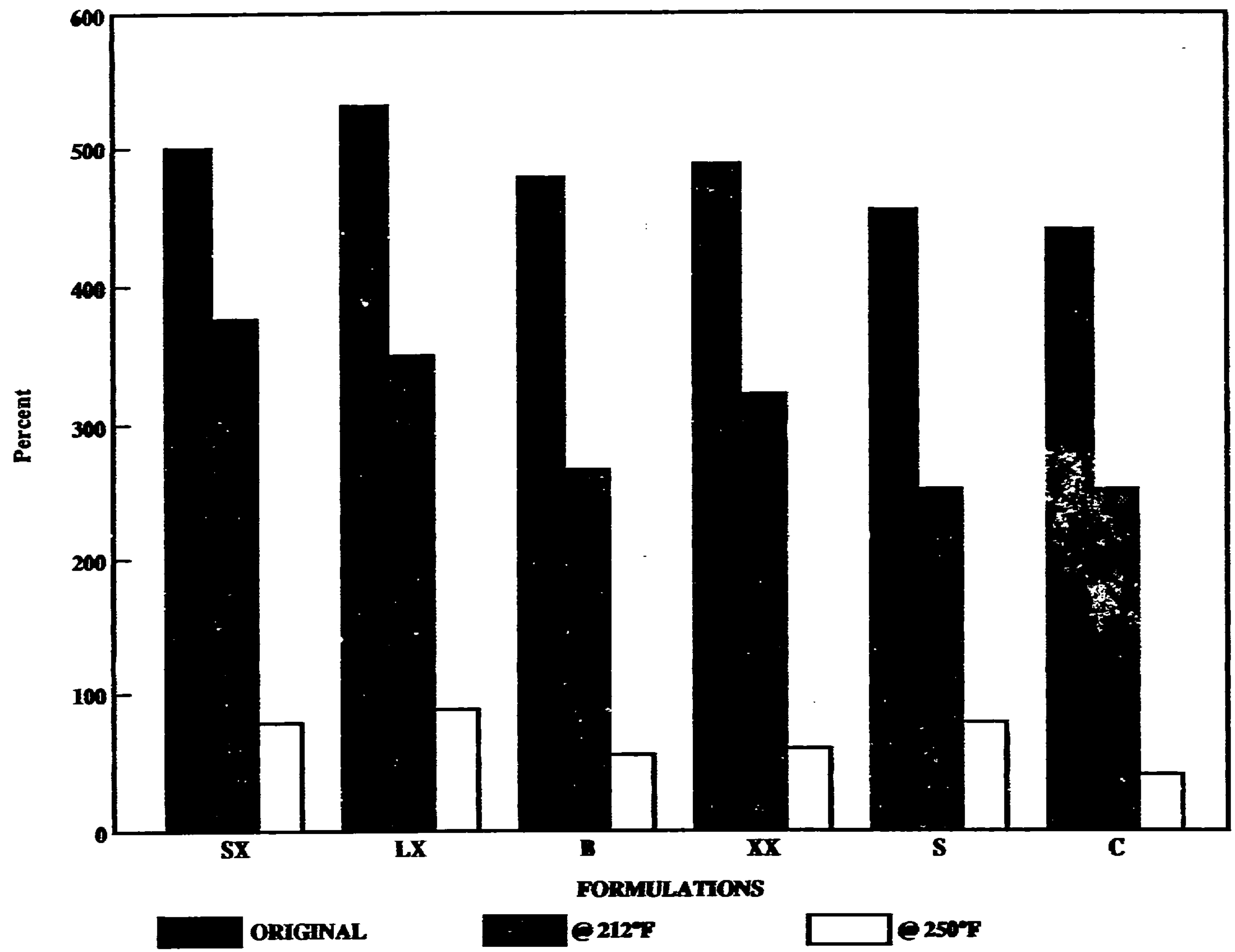


Figure A-17. Ultimate Elongation, Dialkyl and Diaryl PPDs

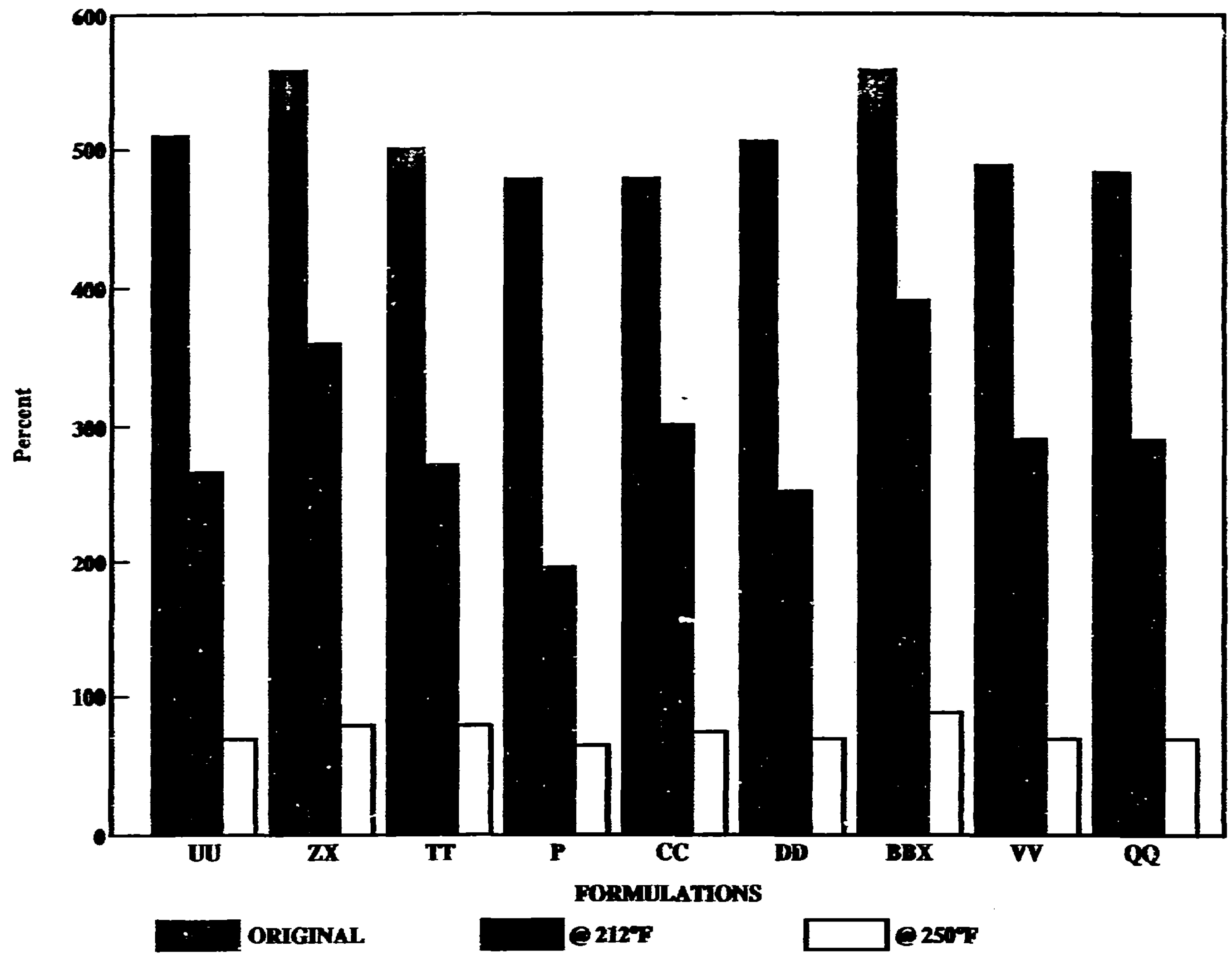


Figure A-18. Ultimate Elongation, Hindered Bisphenols and Phenols

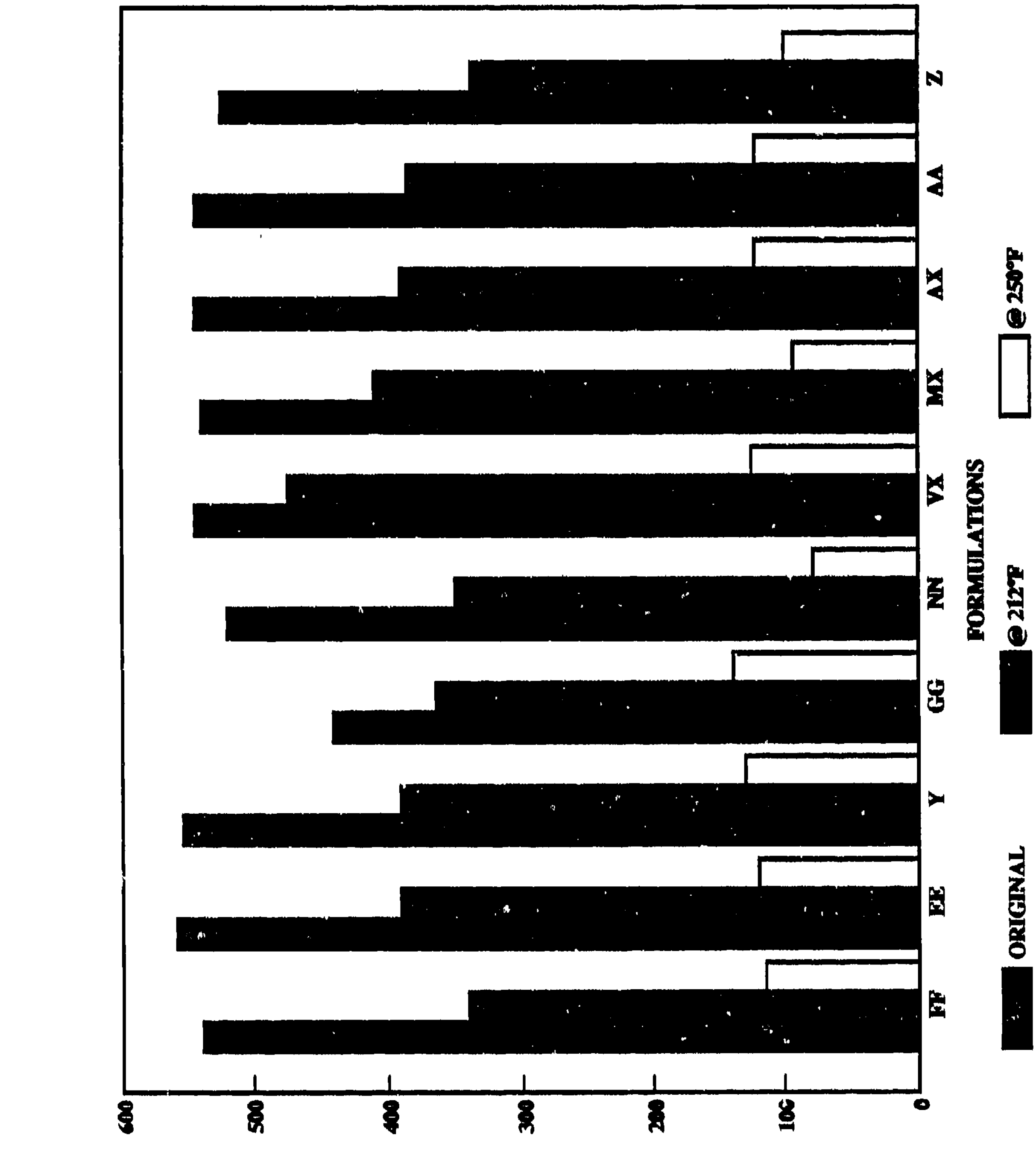


Figure A-19. Ultimate Elongation, MBI, ZMBI, MTI, ZMTI

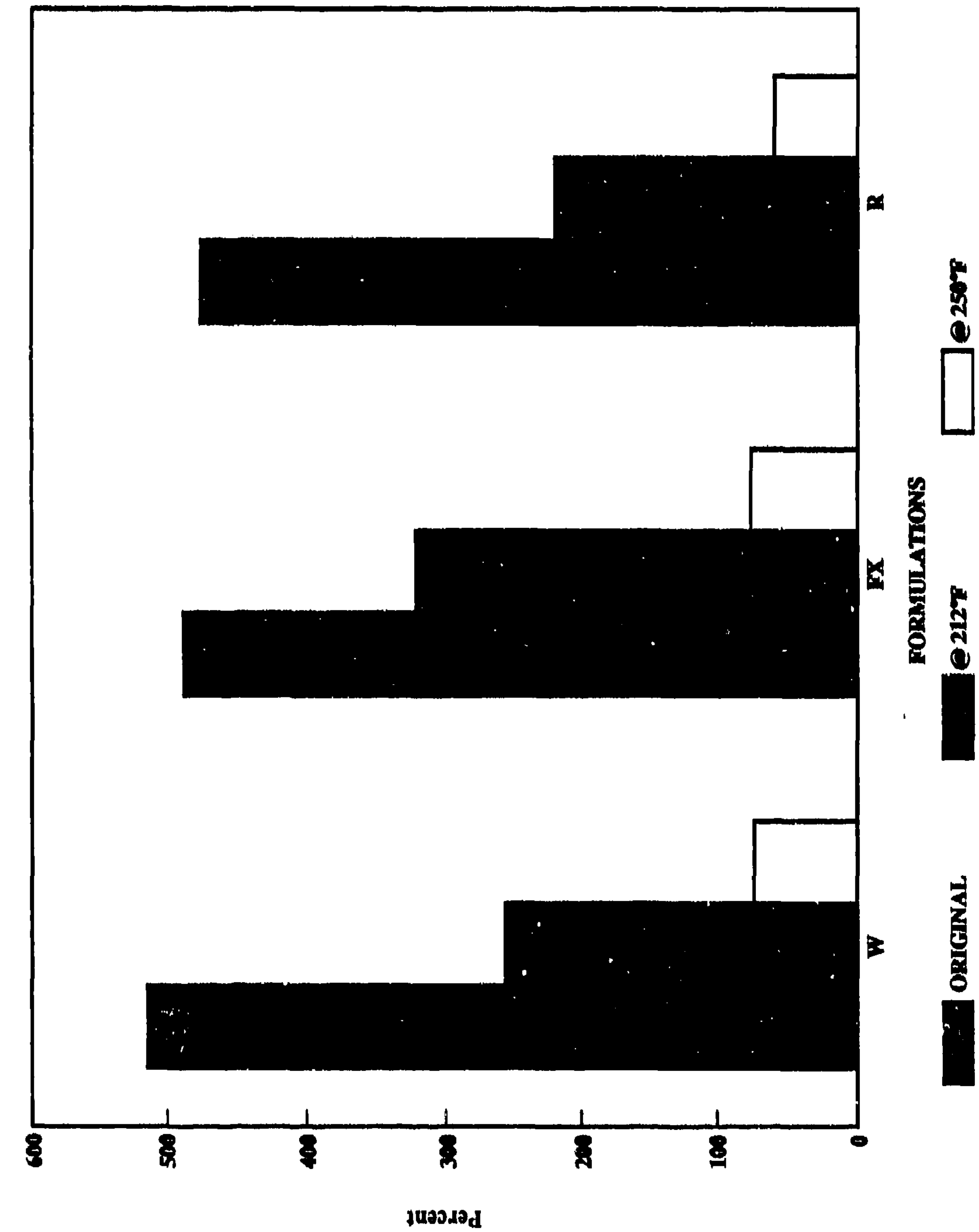


Figure A-20. Ultimate Elongation, Naphthylamines

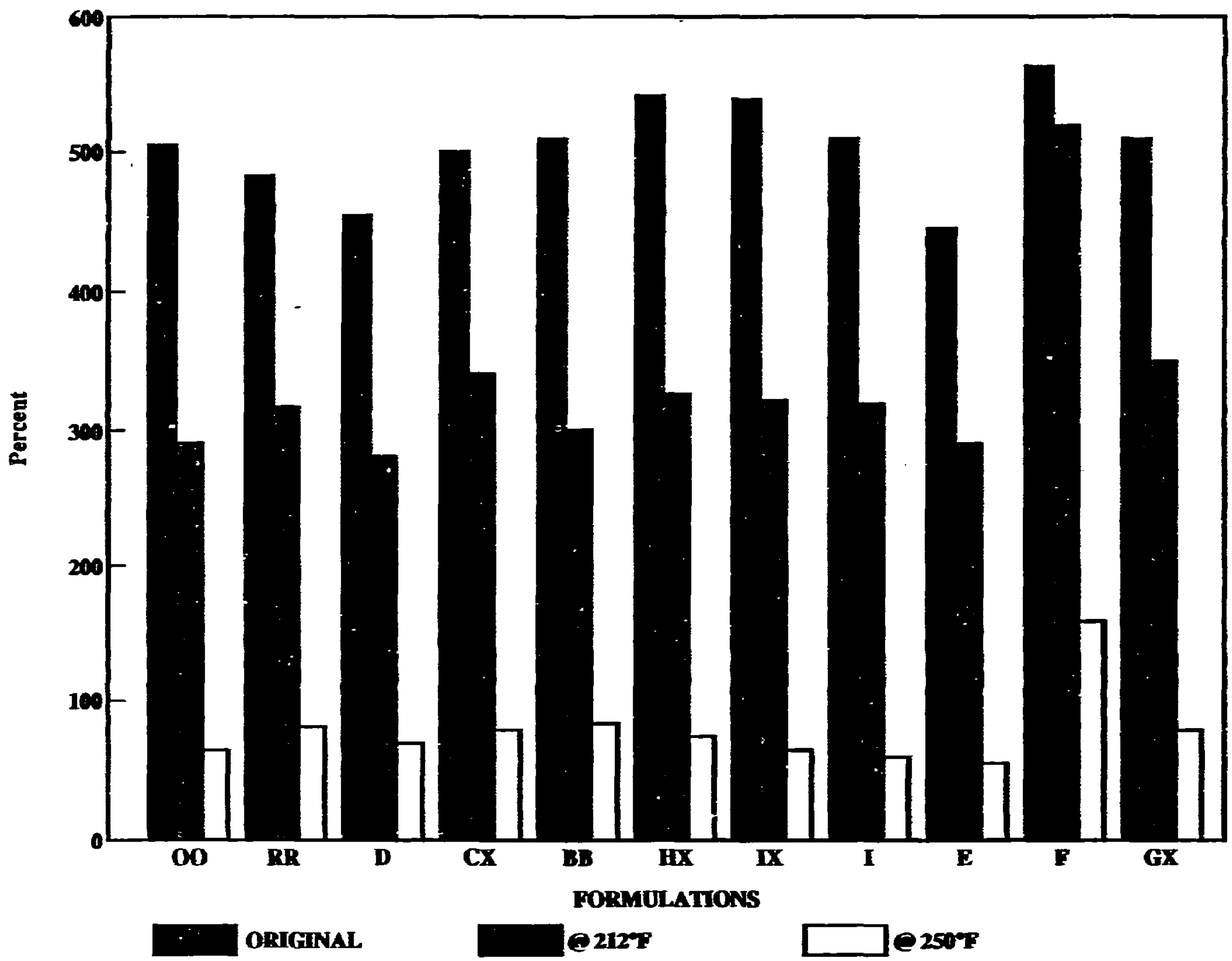


Figure A-21. Ultimate Elongation, Quinolines and Blends

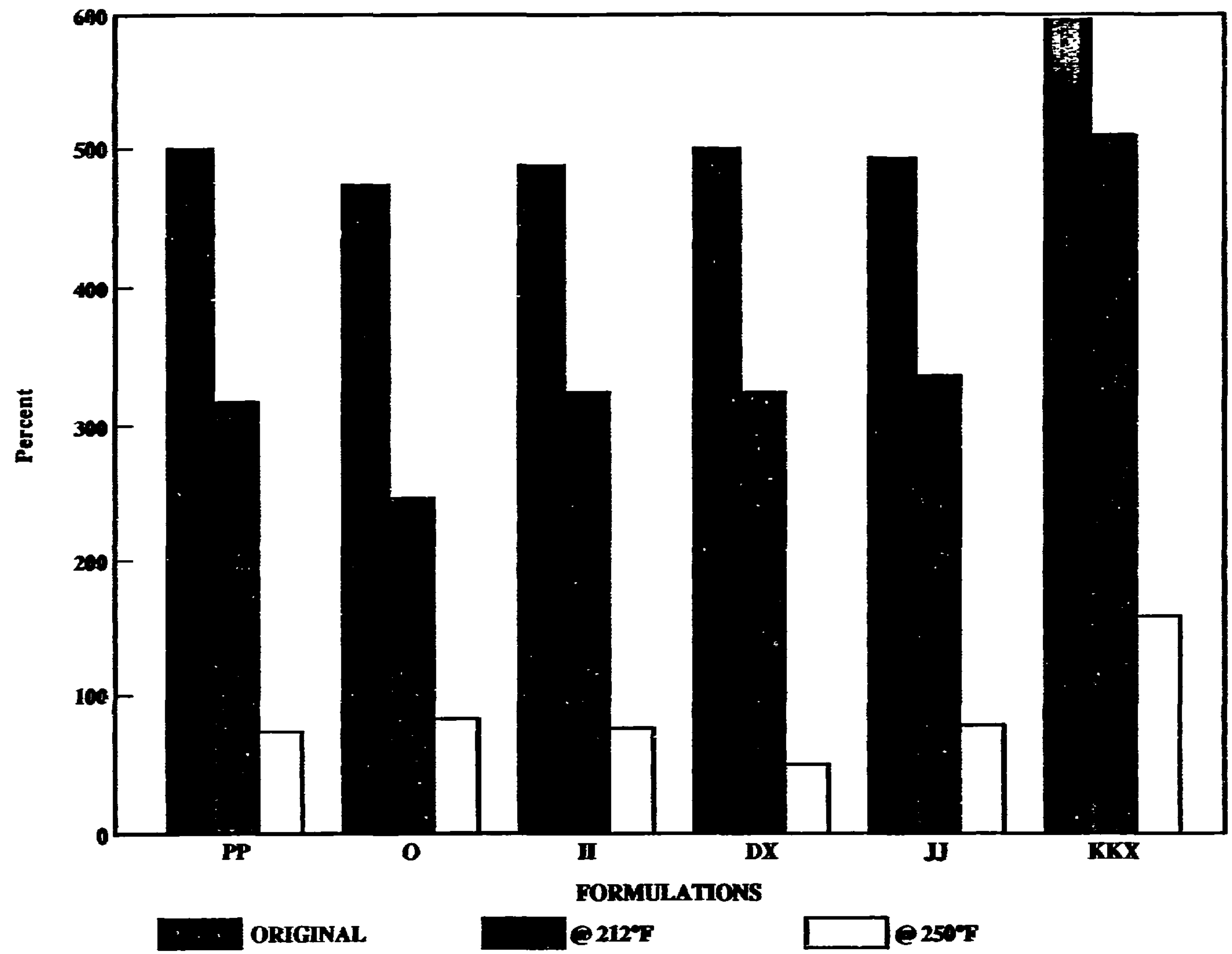


Figure A-22. Ultimate Elongation, Diphenylamines

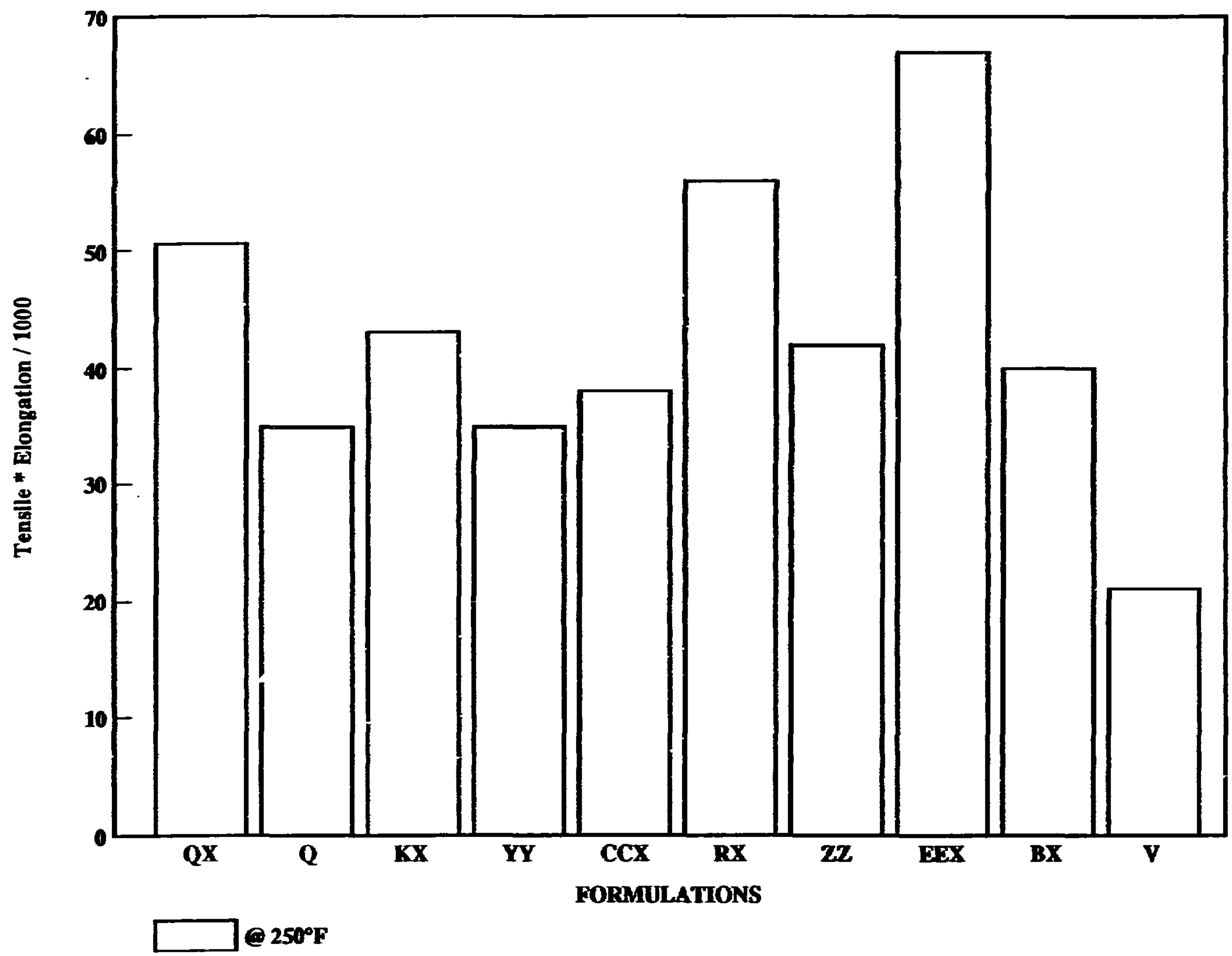


Figure A-23. Tensile * Elongation, Alkyl-Aryl PPDs

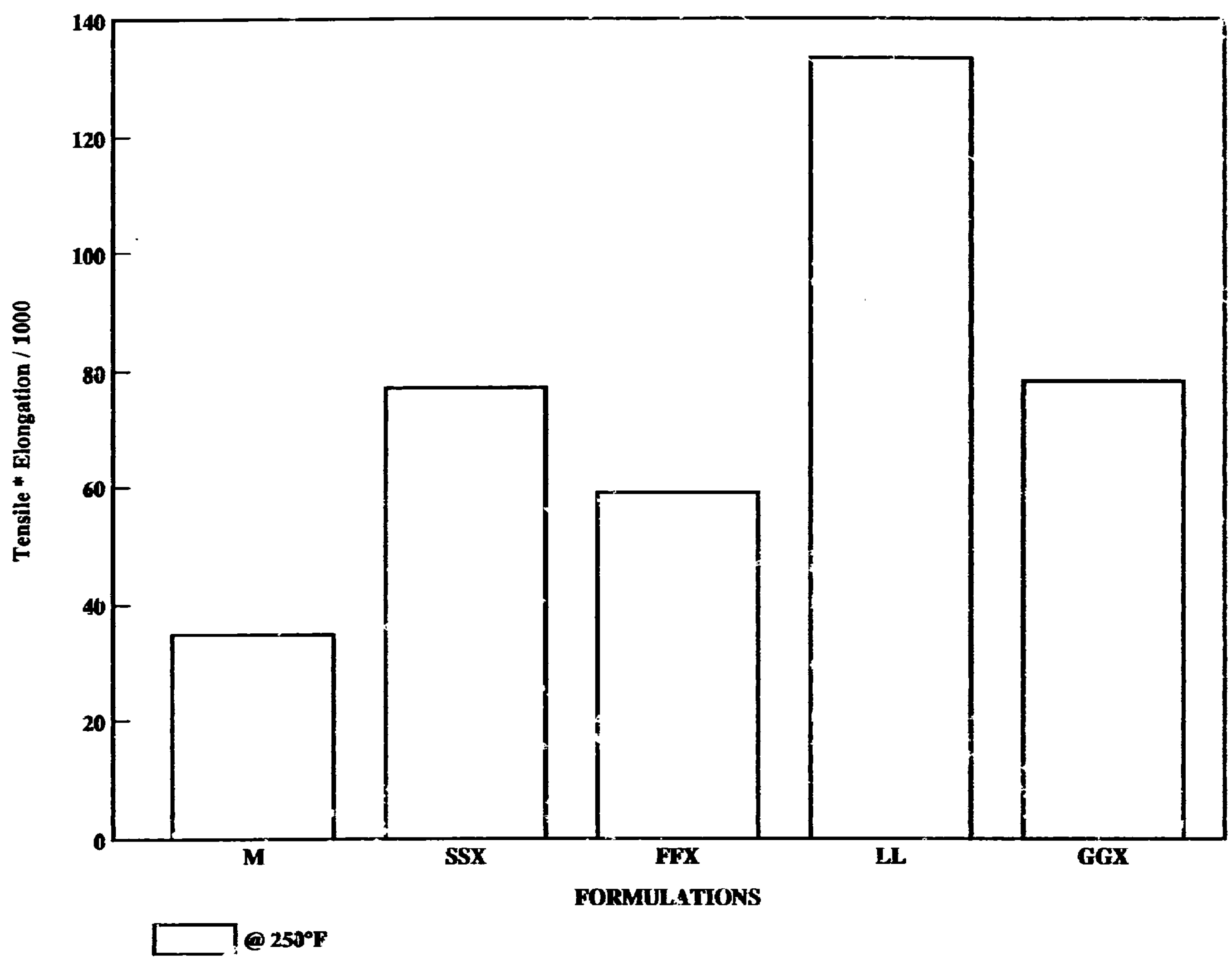


Figure A-24. Tensile * Elongation, Alkyl-Aryl PPDs/Quinolines

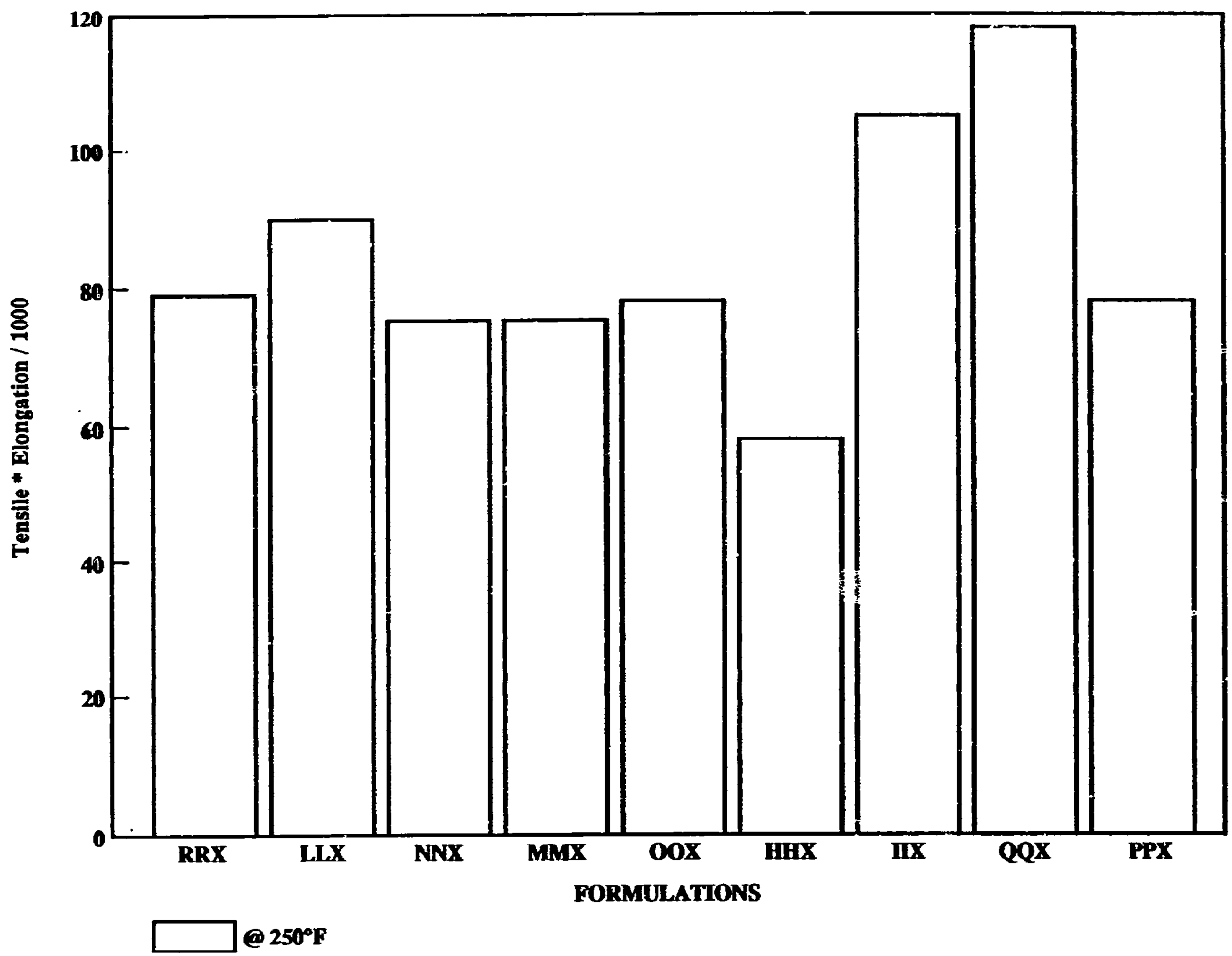


Figure A-25. Tensile * Elongation, Alkyl-Aryl PPDs/Quinolines/Others

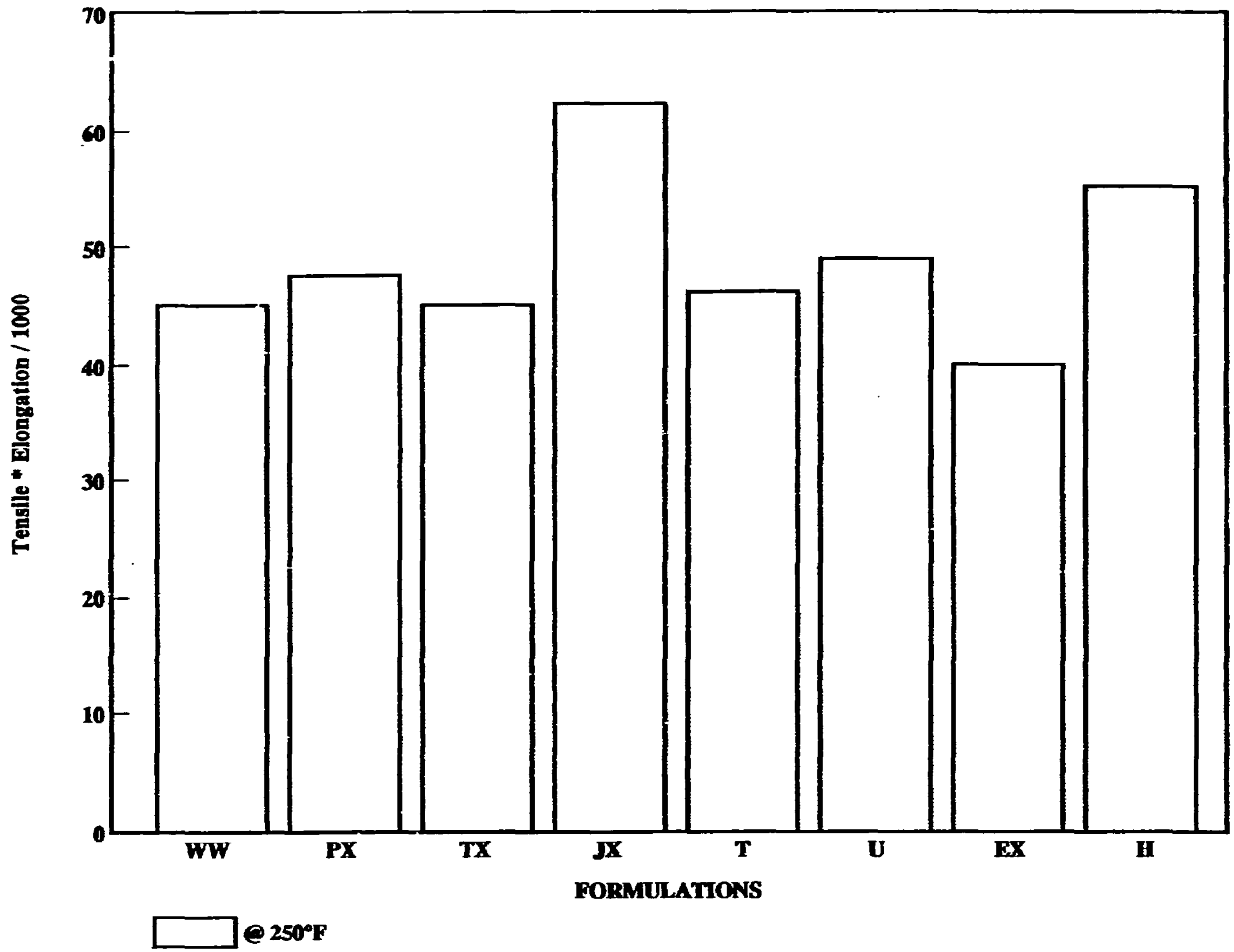


Figure A-26. Tensile * Elongation, Blended Amines

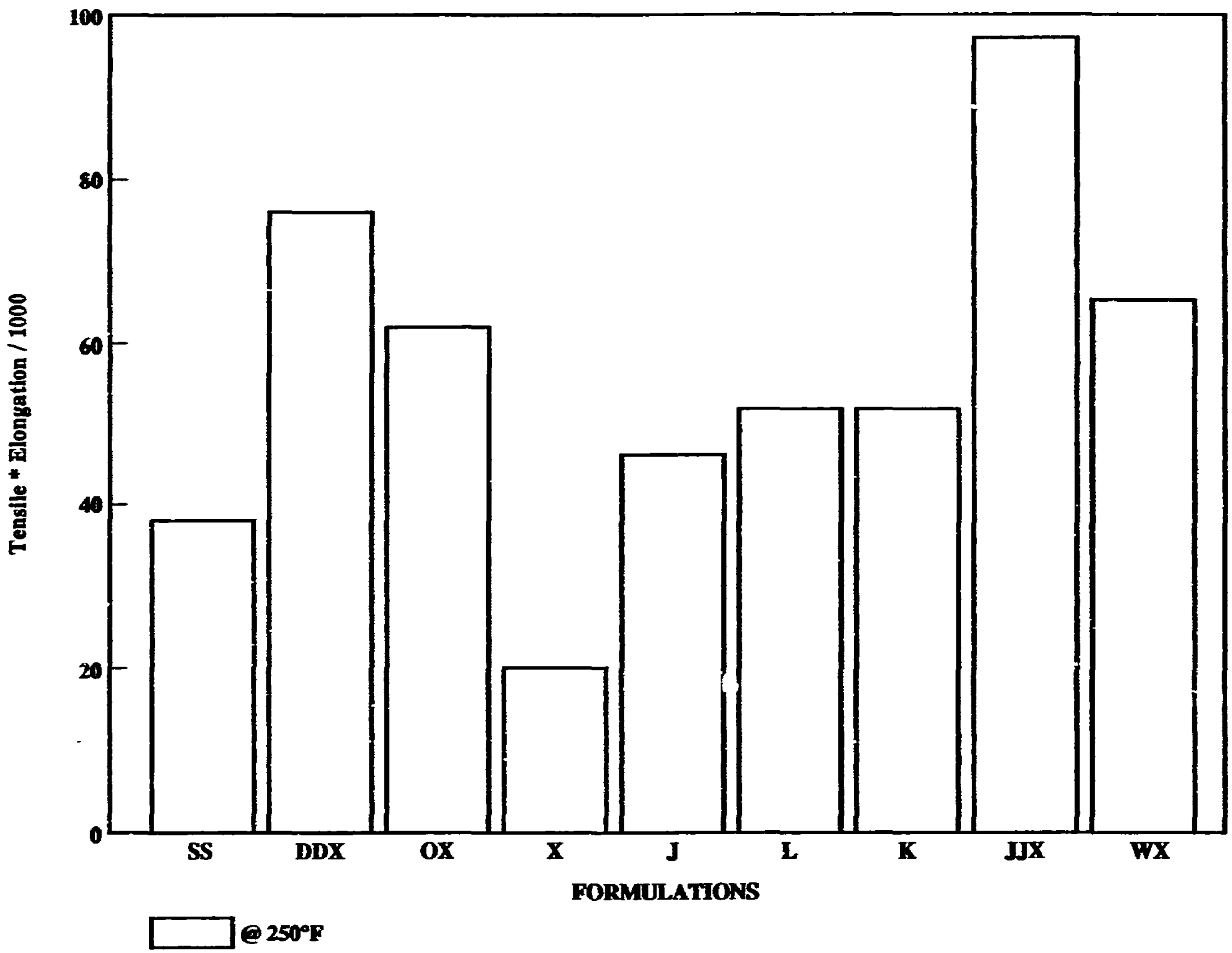


Figure A-27. Tensile * Elongation, Condensation Products

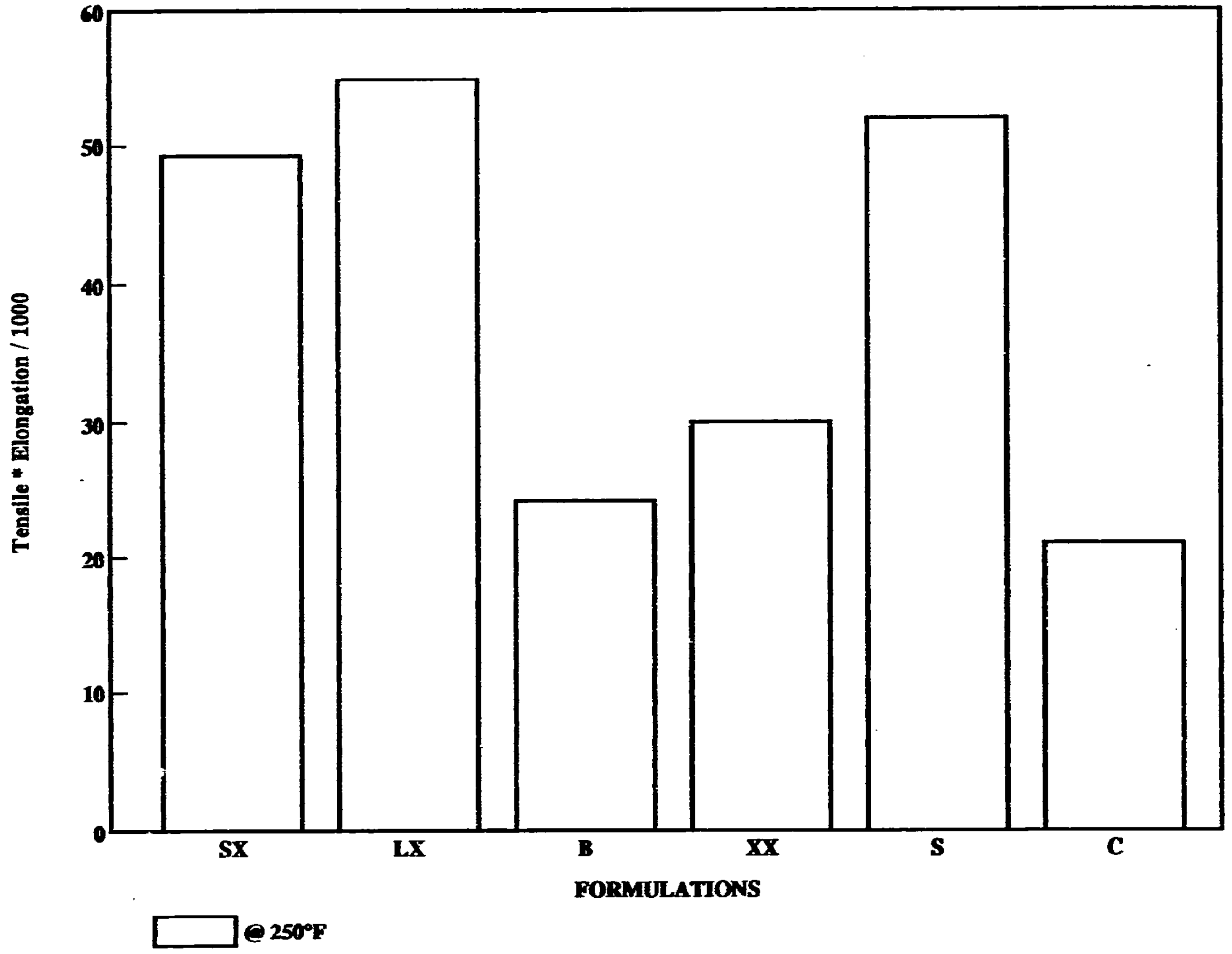


Figure A-28. Tensile * Elongation, Dialkyl and Diaryl PPDs

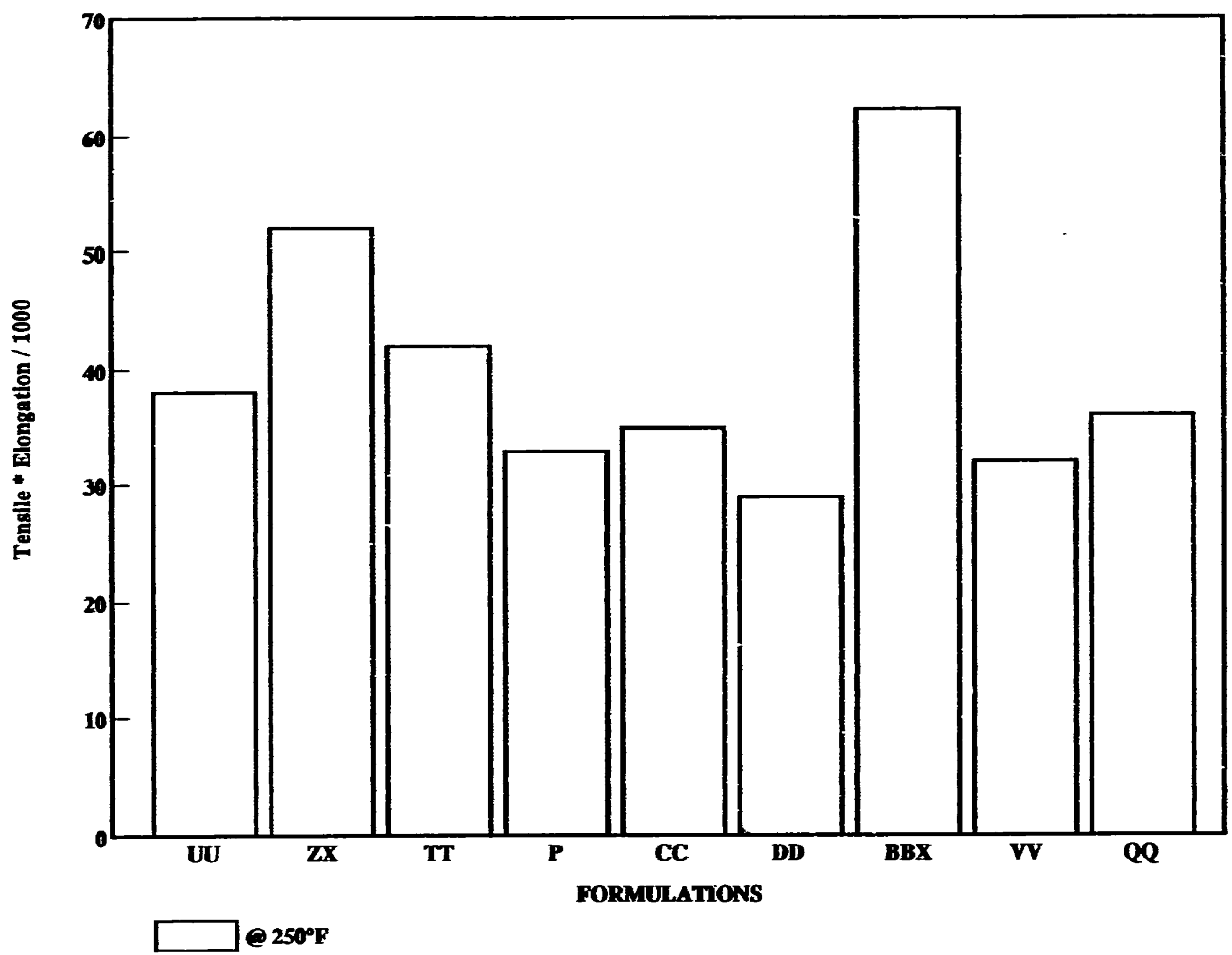


Figure A-29. Tensile * Elongation, Hindered Bisphenols and Phenols

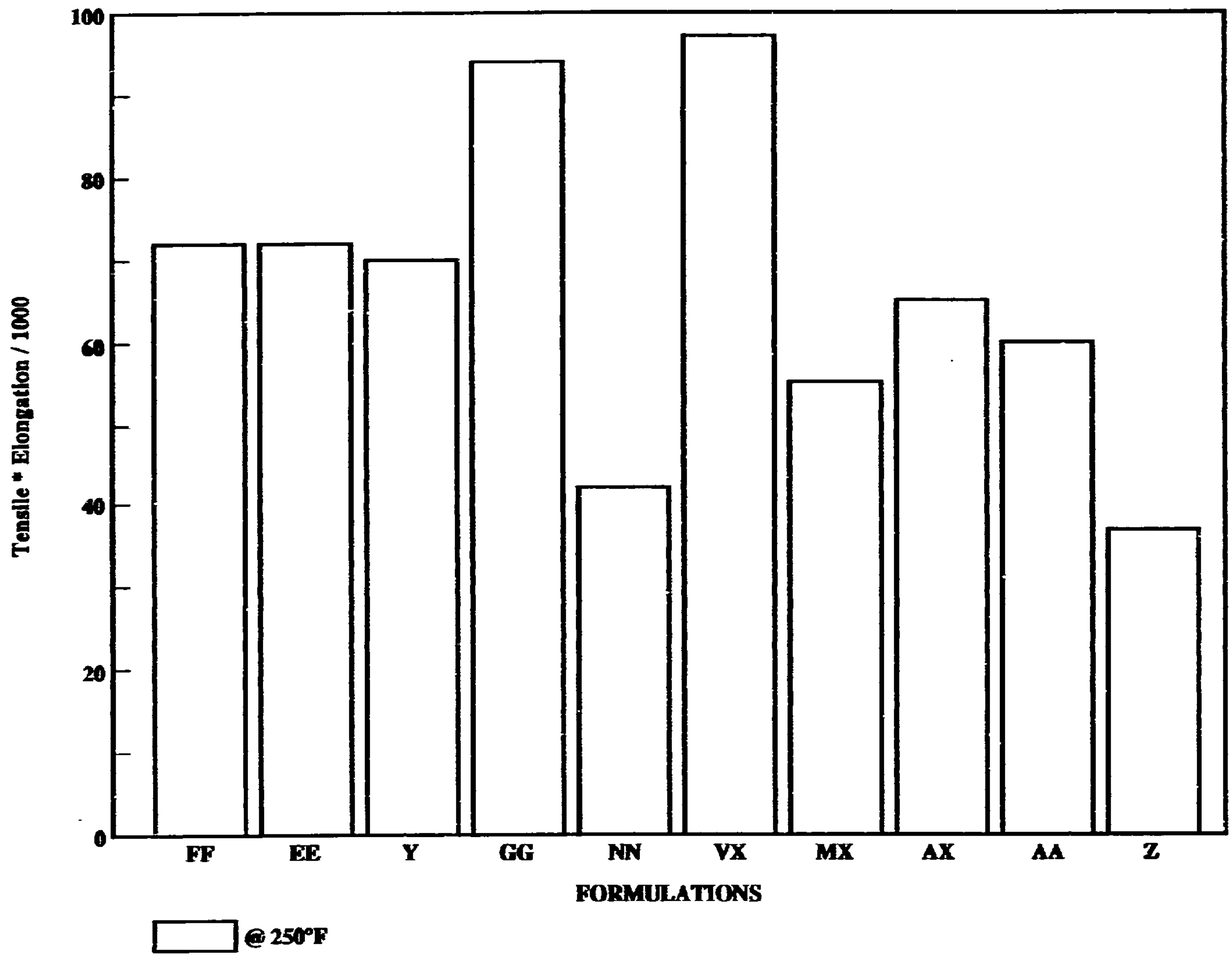


Figure A-30. Tensile * Elongation, MBI, ZMBI, MTI, ZMTI

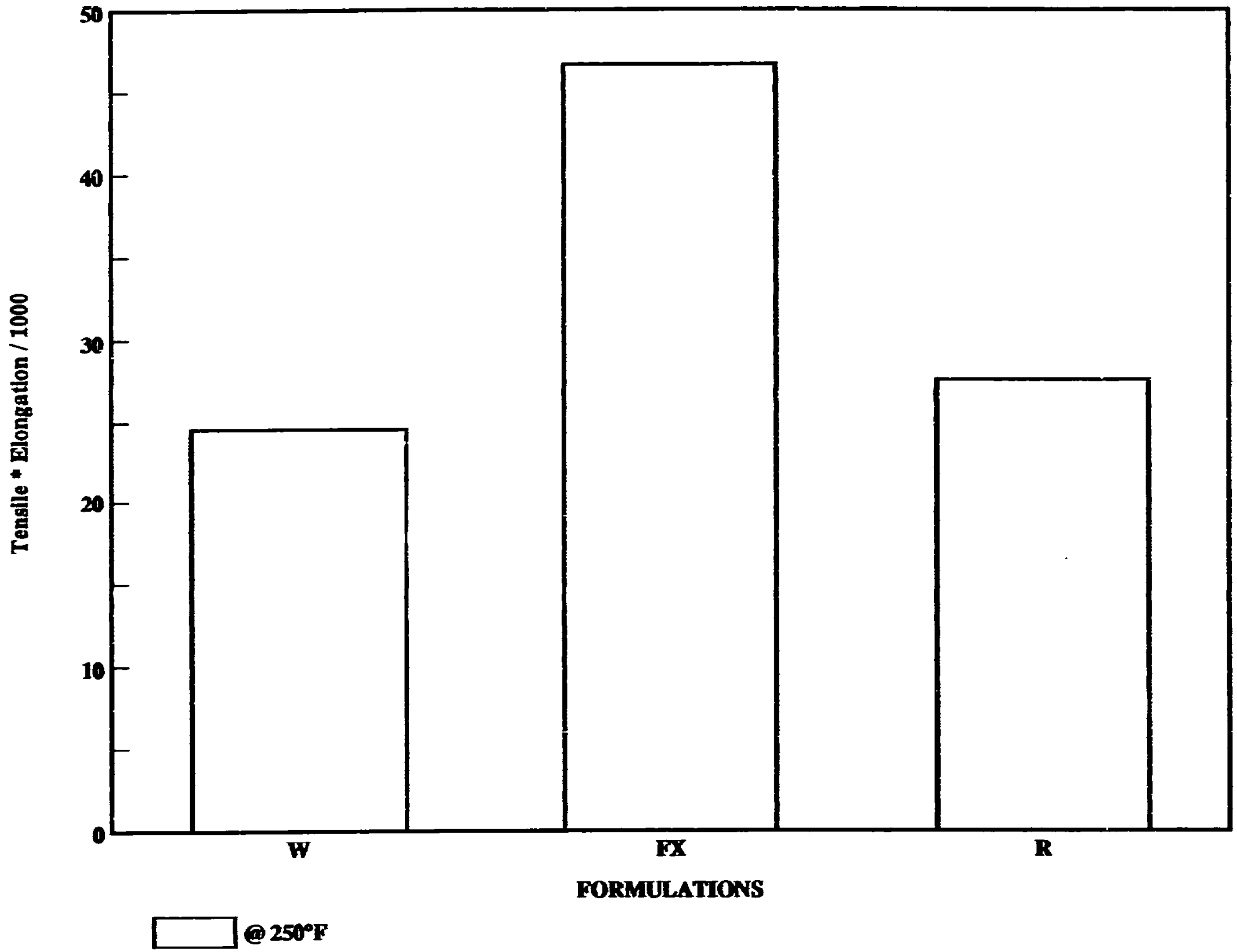


Figure A-31. Tensile * Elongation, Naphthylamines

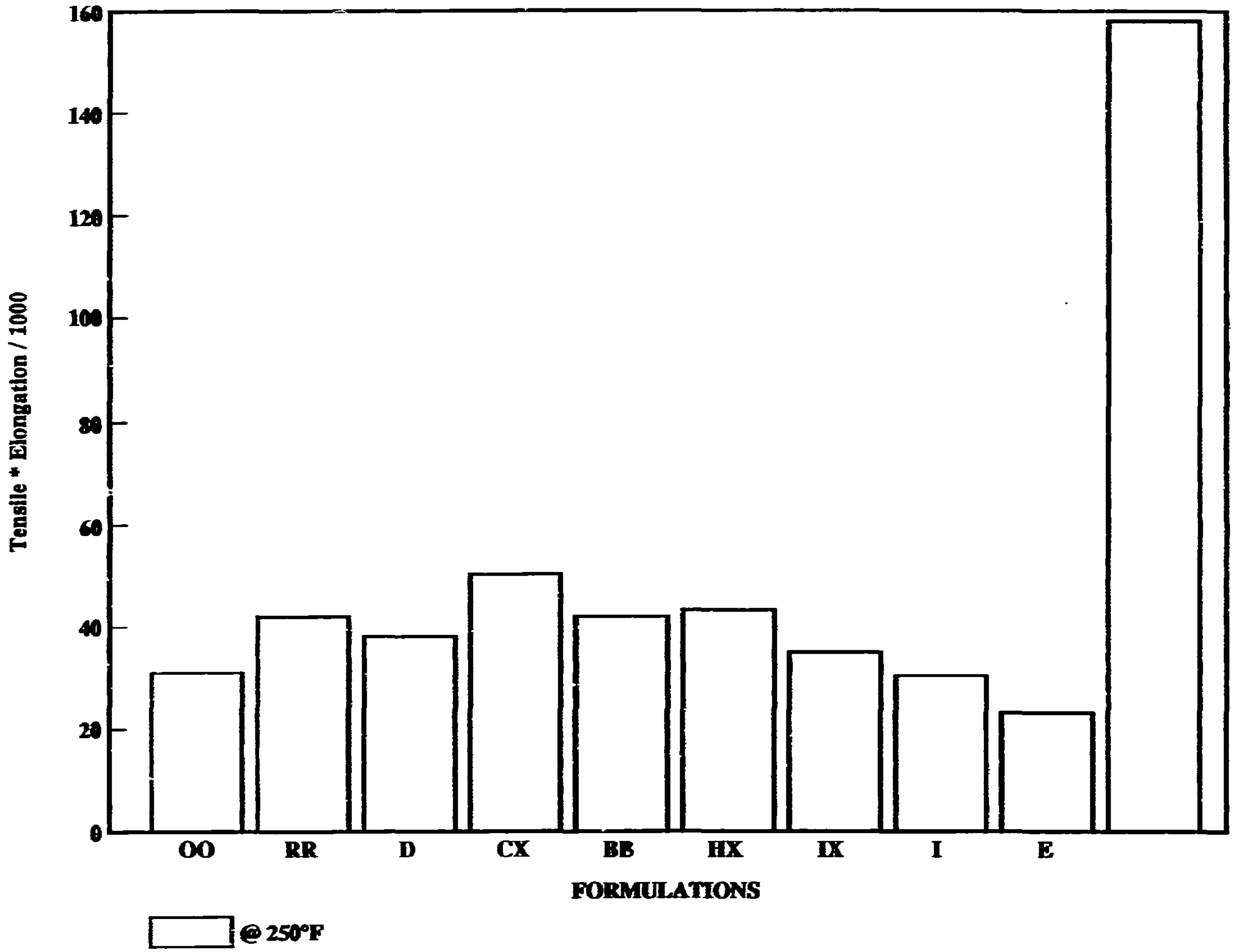


Figure A-32. Tensile * Elongation, Quinolines and Mixtures

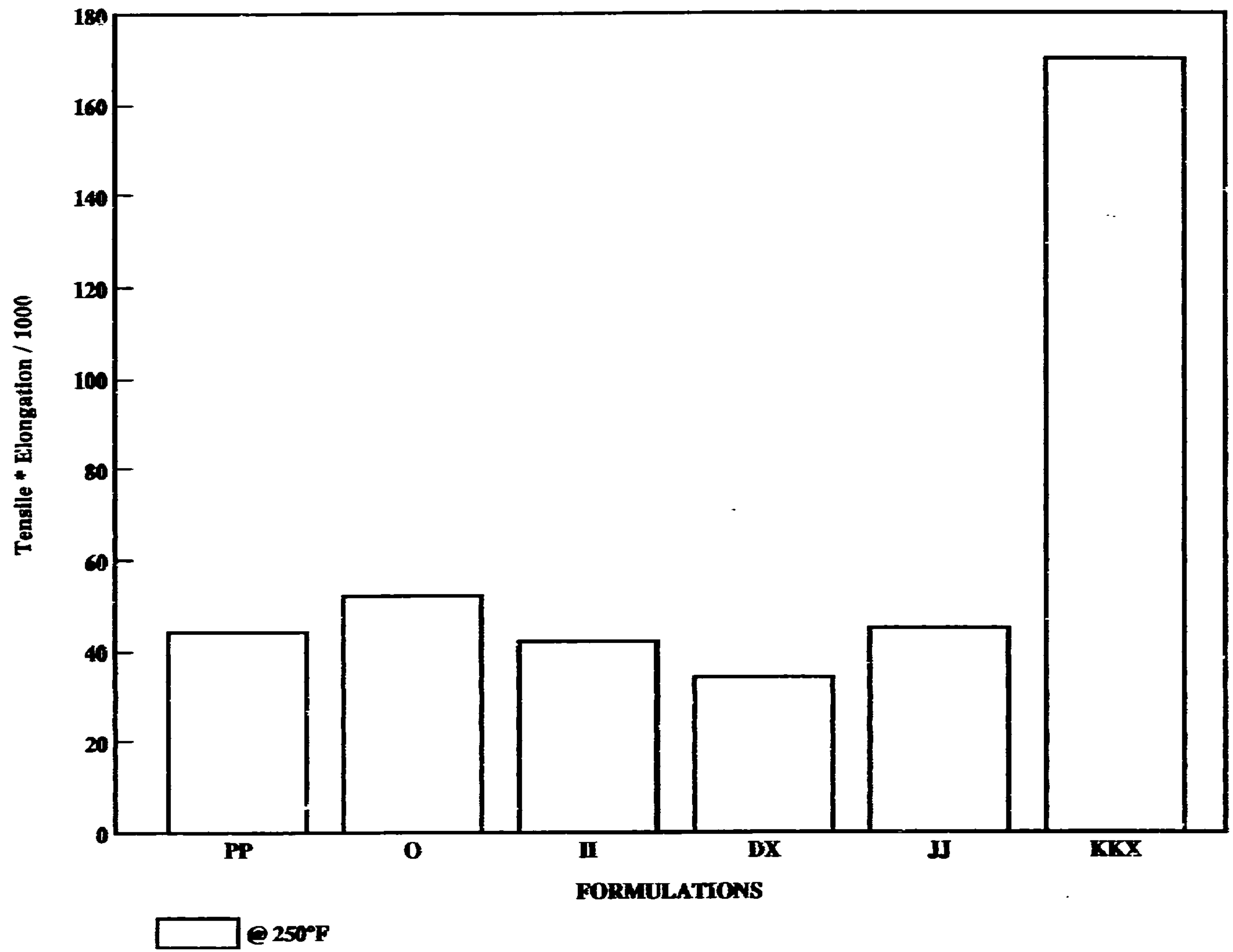


Figure A-33. Tensile * Elongation, Dyphenylamines

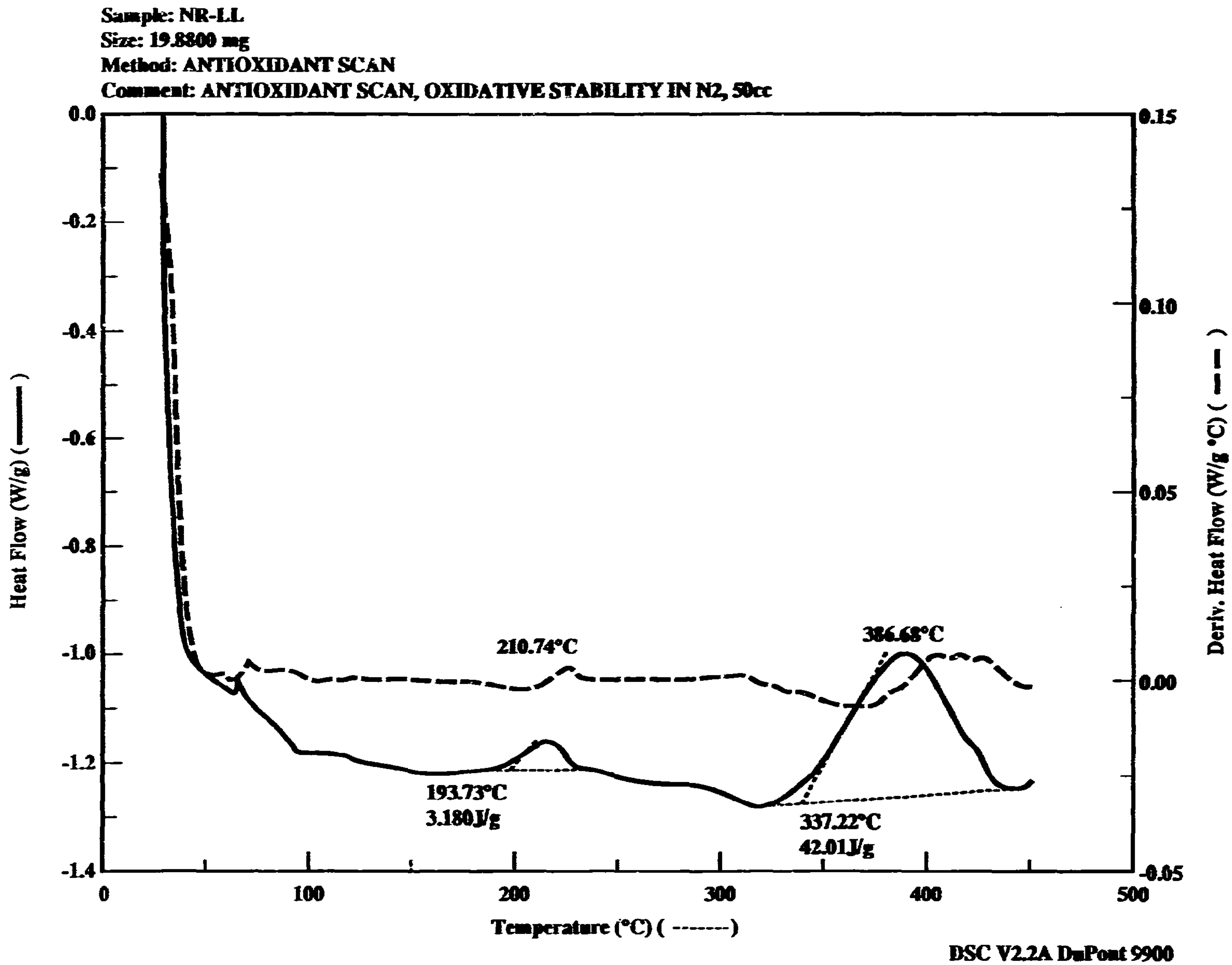


Figure A-34. DSC, NR-LL

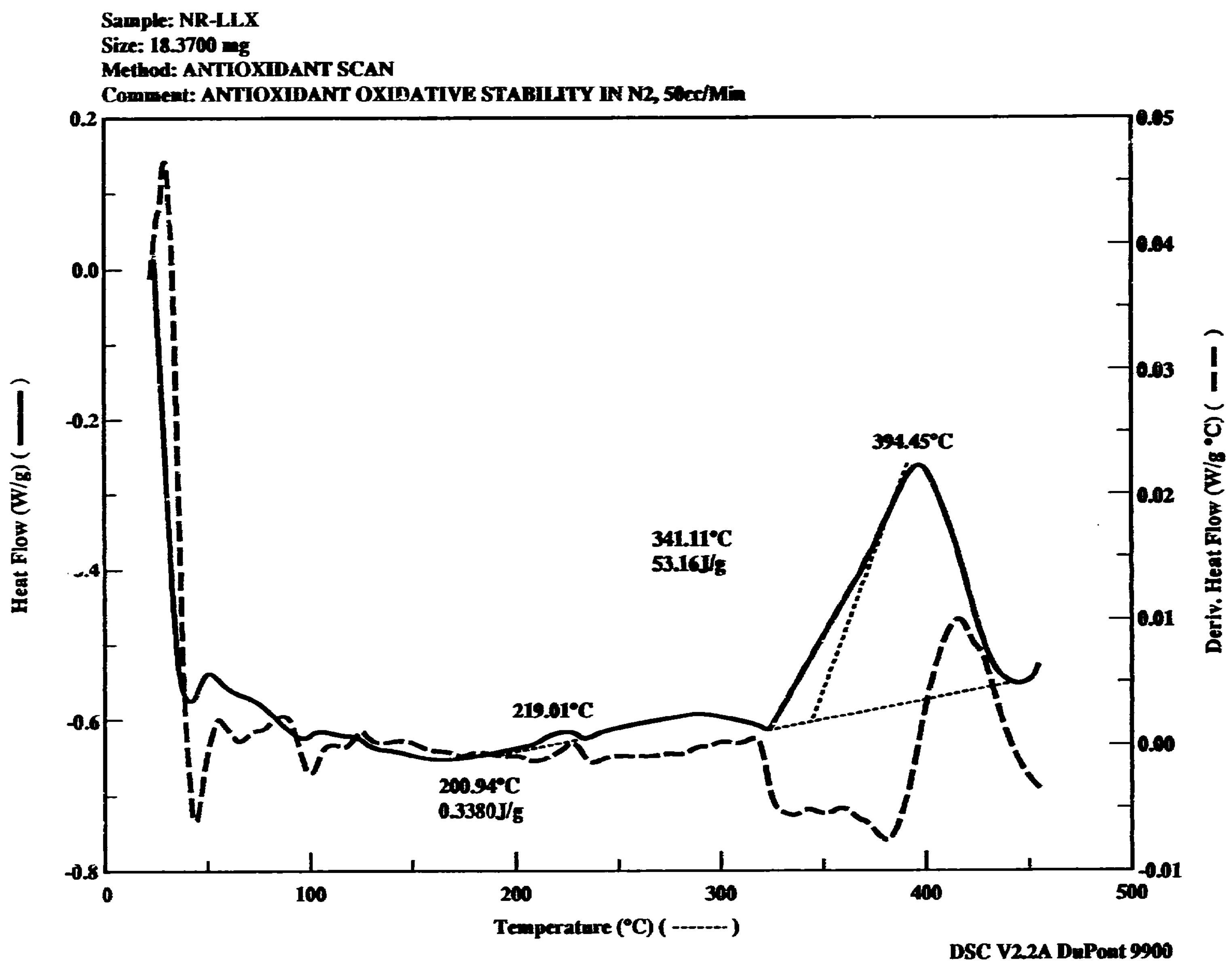


Figure A-35. DSC, NR-LLX

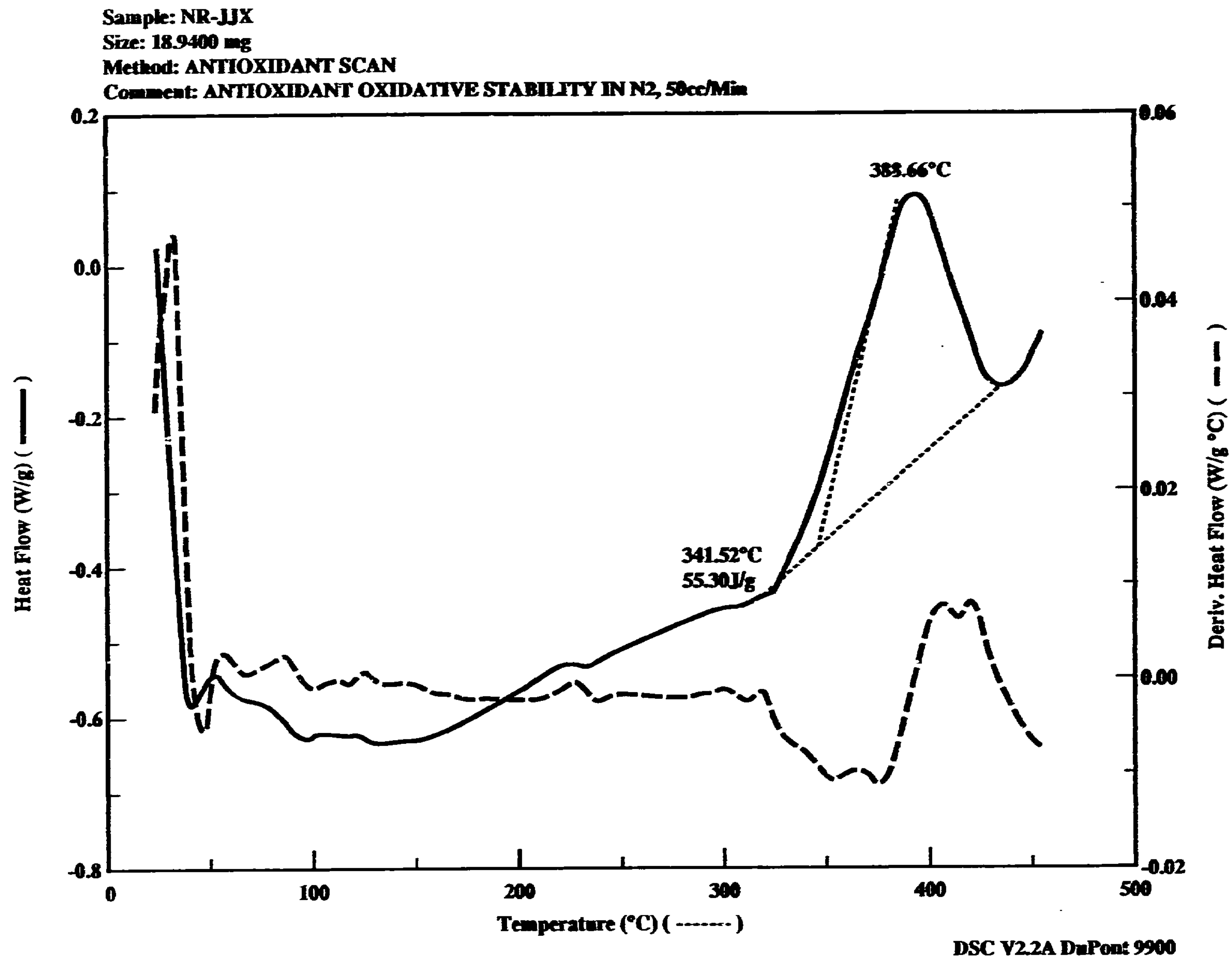


Figure A-36. DSC, NR-JJX

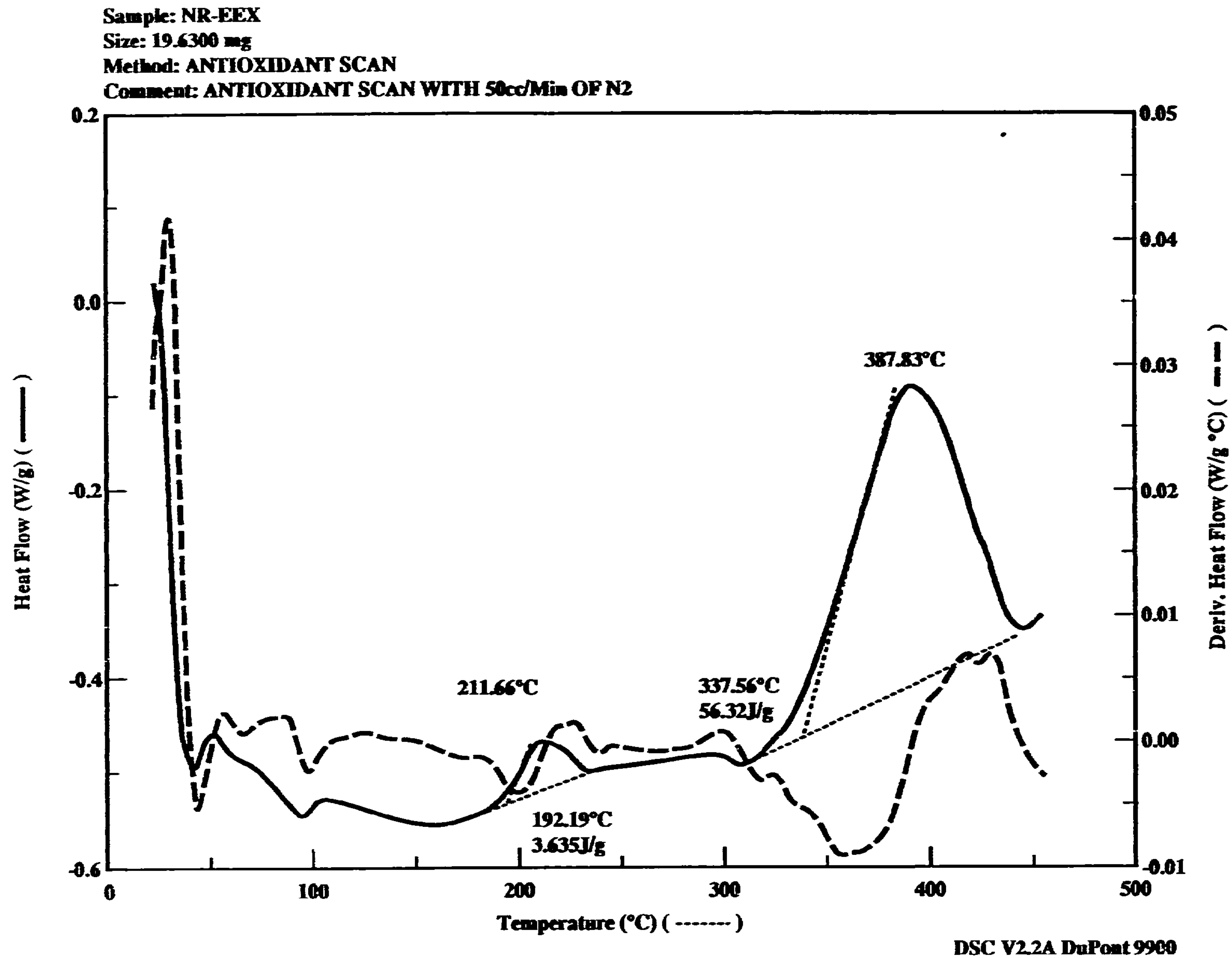
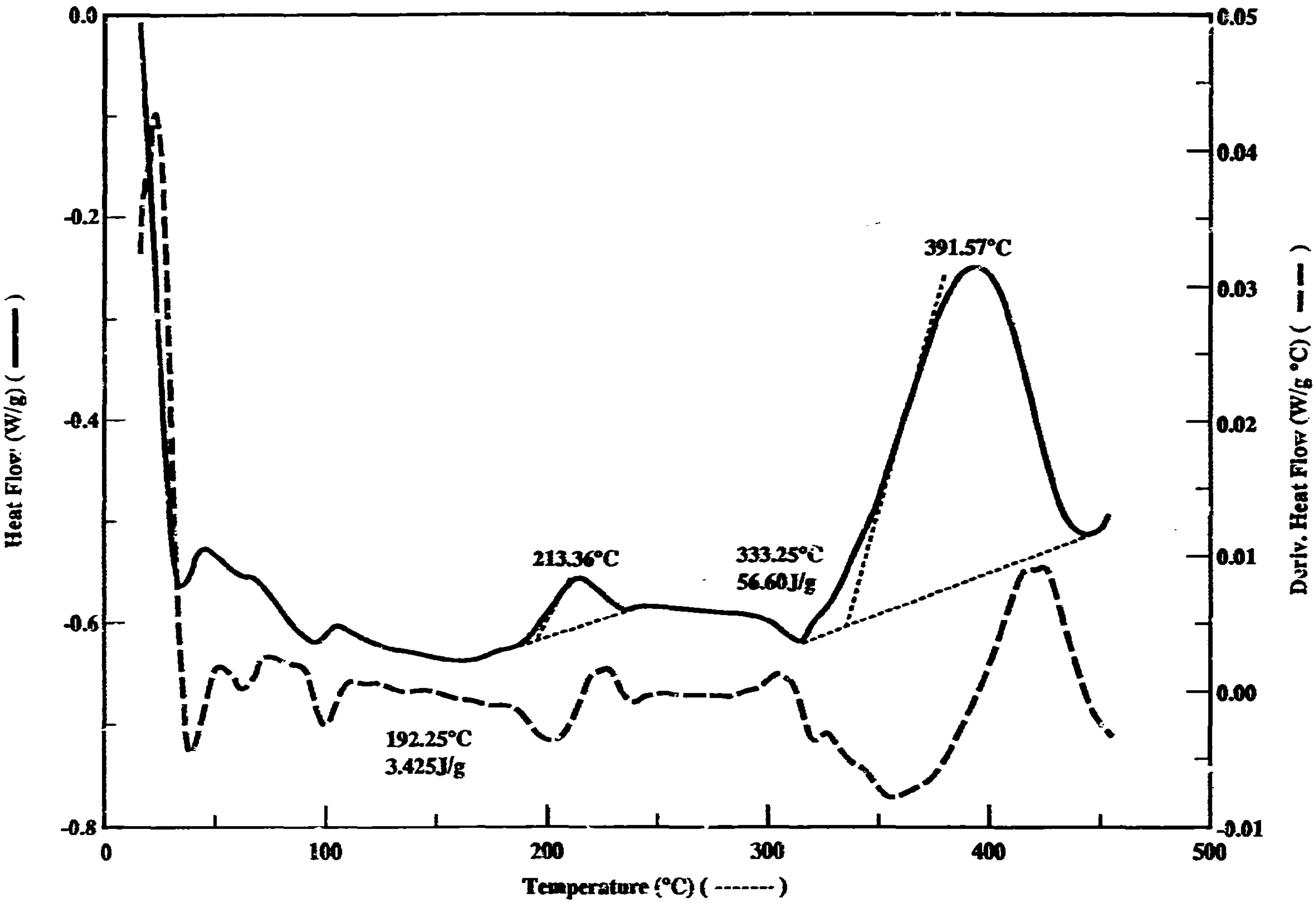


Figure A-37. DSC, NR-EEX

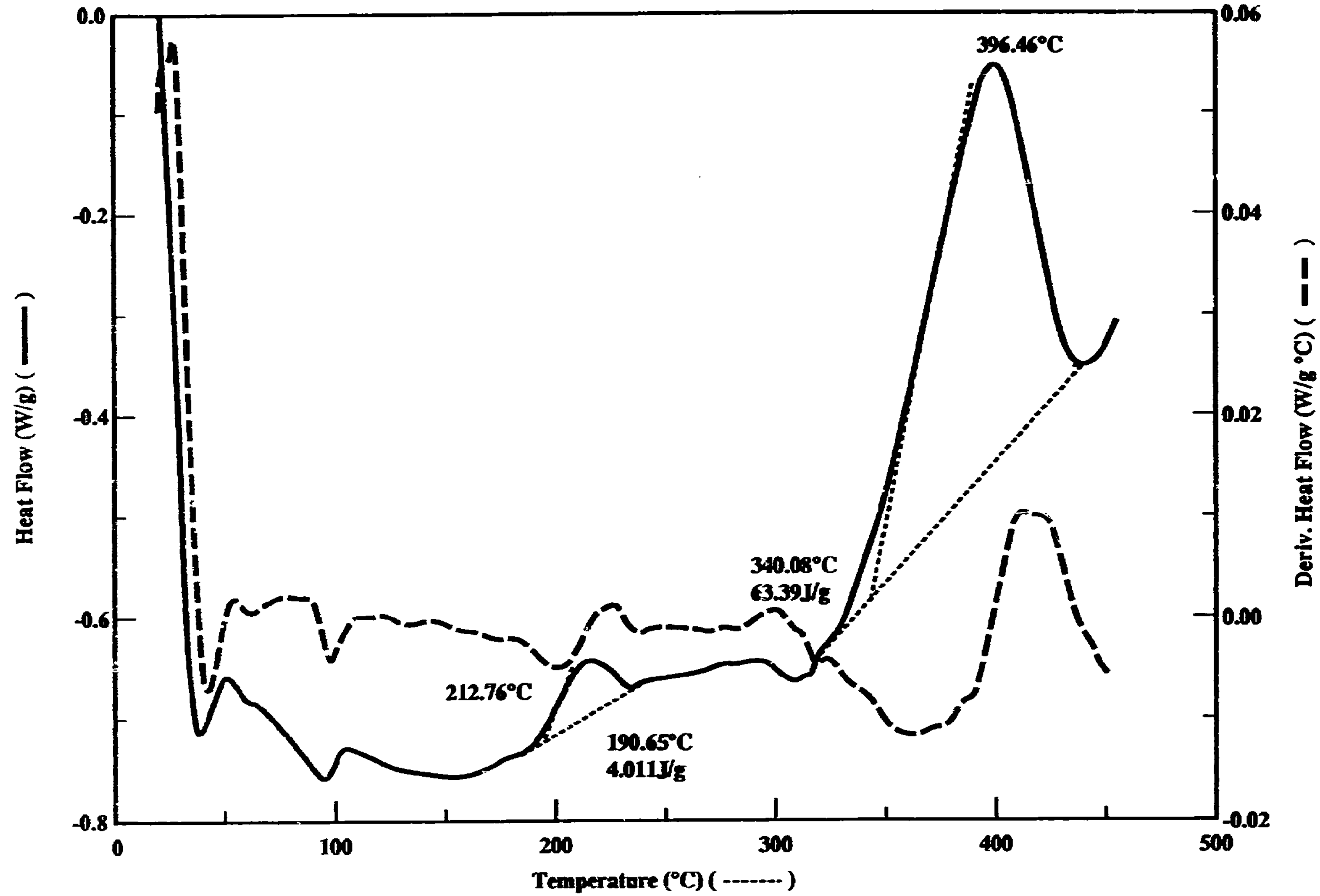
Sample: NR-RRX
Size: 18.5400 mg
Method: ANTIOXIDANT SCAN
Comment: ANTIOXIDANT OXIDATIVE STABILITY IN N2, 50cc/Min



DSC V2.2A DuPont 9900

Figure A-38. DSC, NR-RRX

Sample: NP.-HHX
Size: 17.9500 mg
Method: ANTIOXIDANT SCAN
Comment: ANTIOXIDANT OXIDATIVE STABILITY IN N2, 50cc/Min



DSC V2.2A DuPont 9900

Figure A-39. DSC, NR-HHX

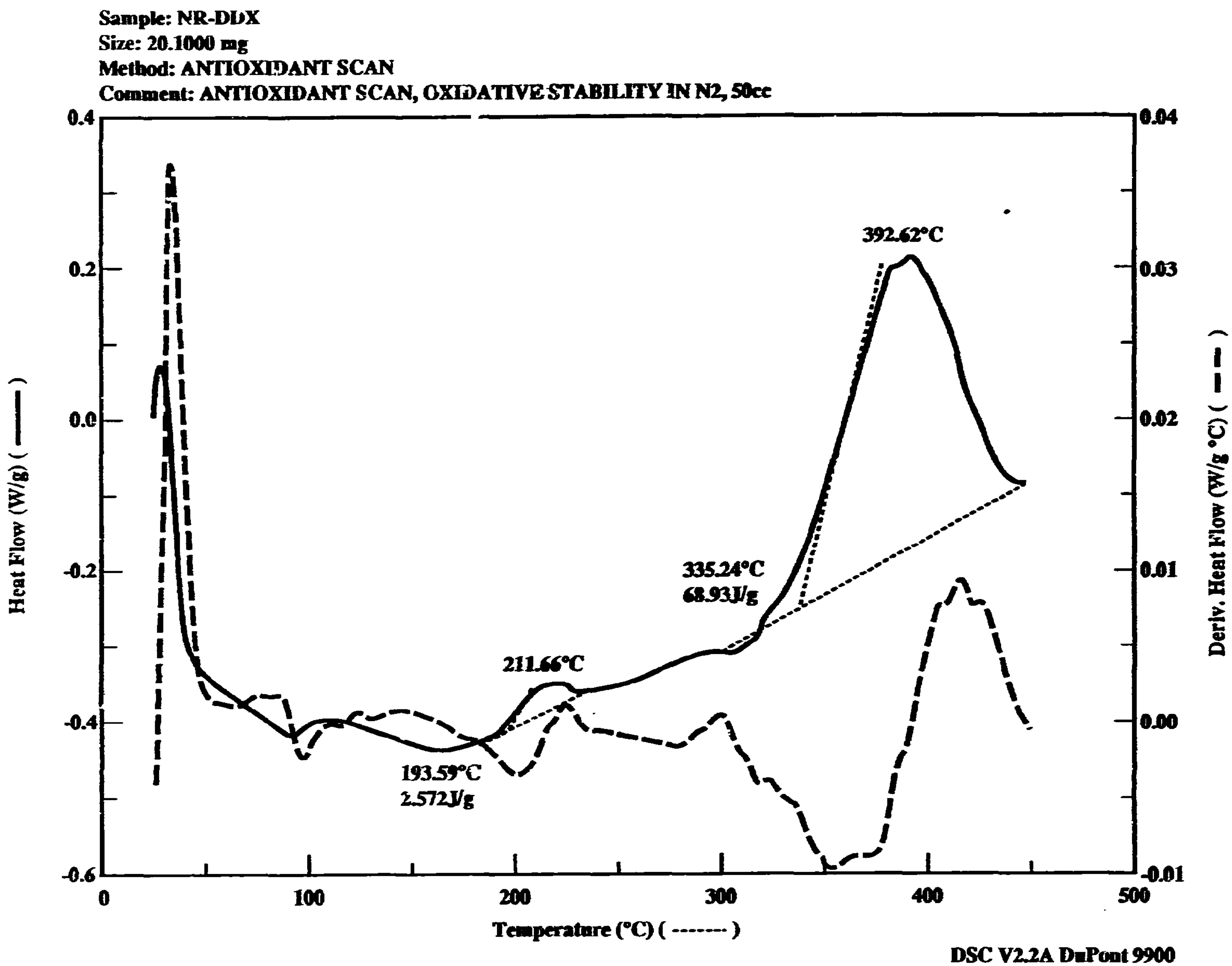


Figure A-40. DSC, NR-DDX

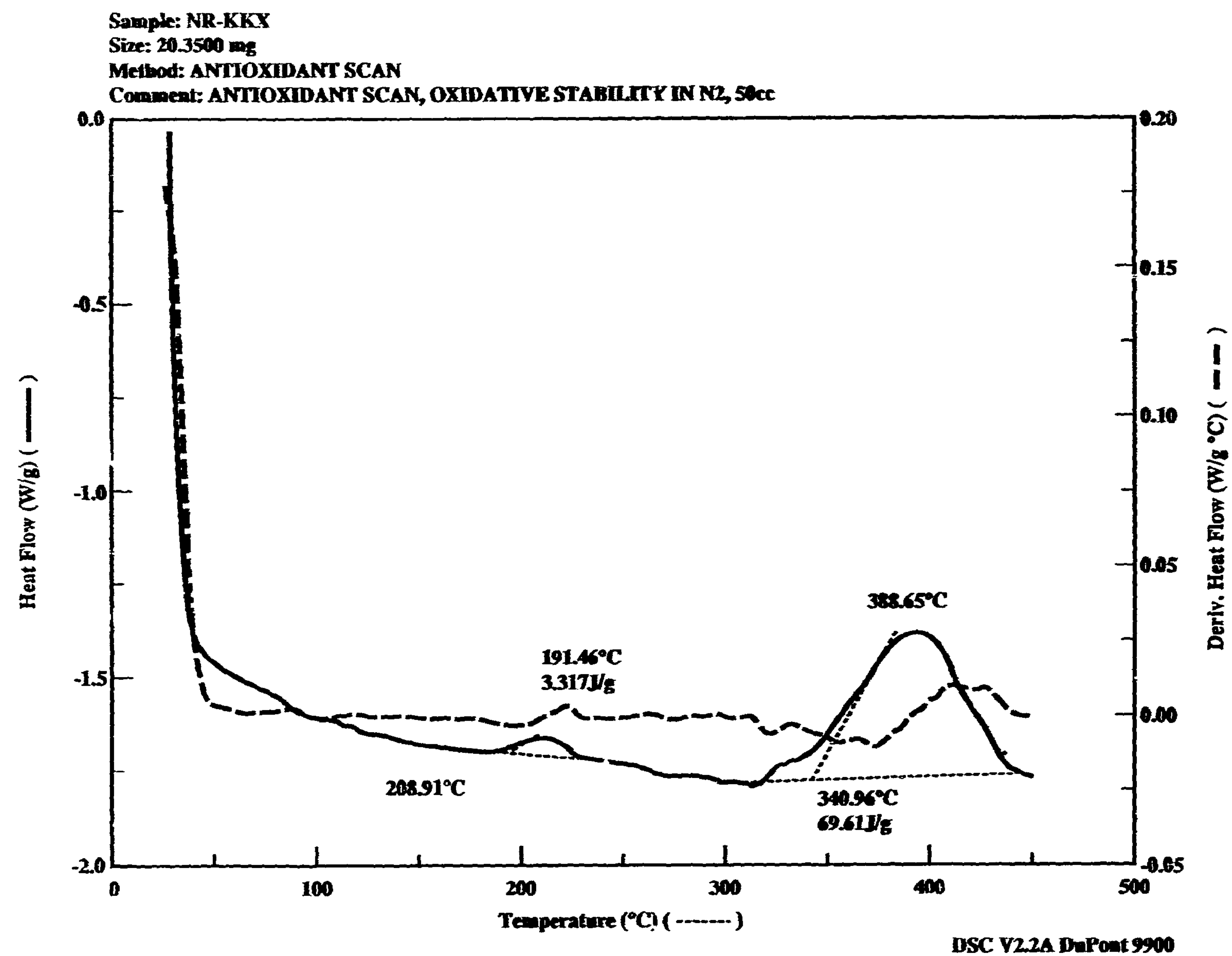


Figure A-41. DSC, NR-KKX

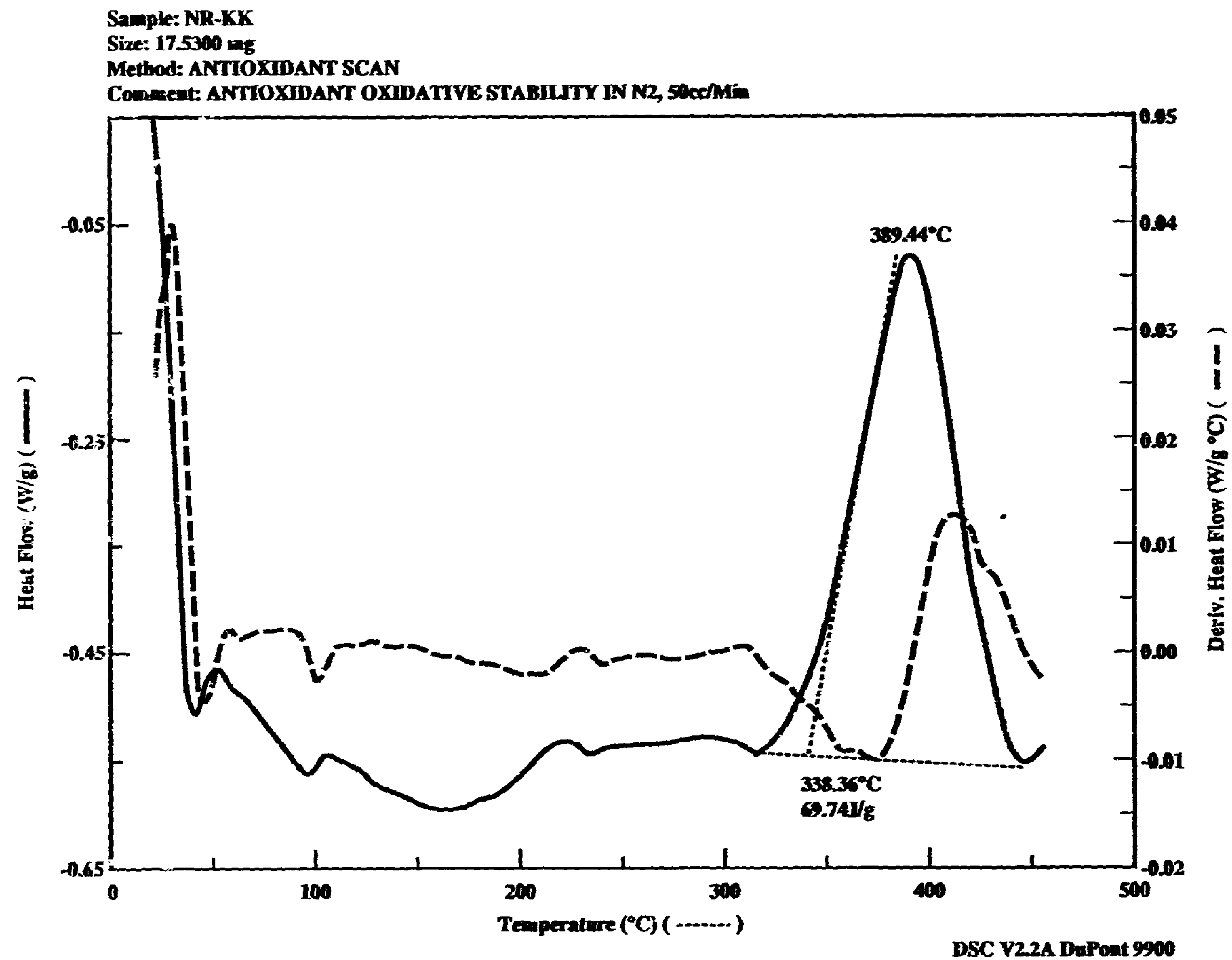


Figure A-42. DSC, NR-KK

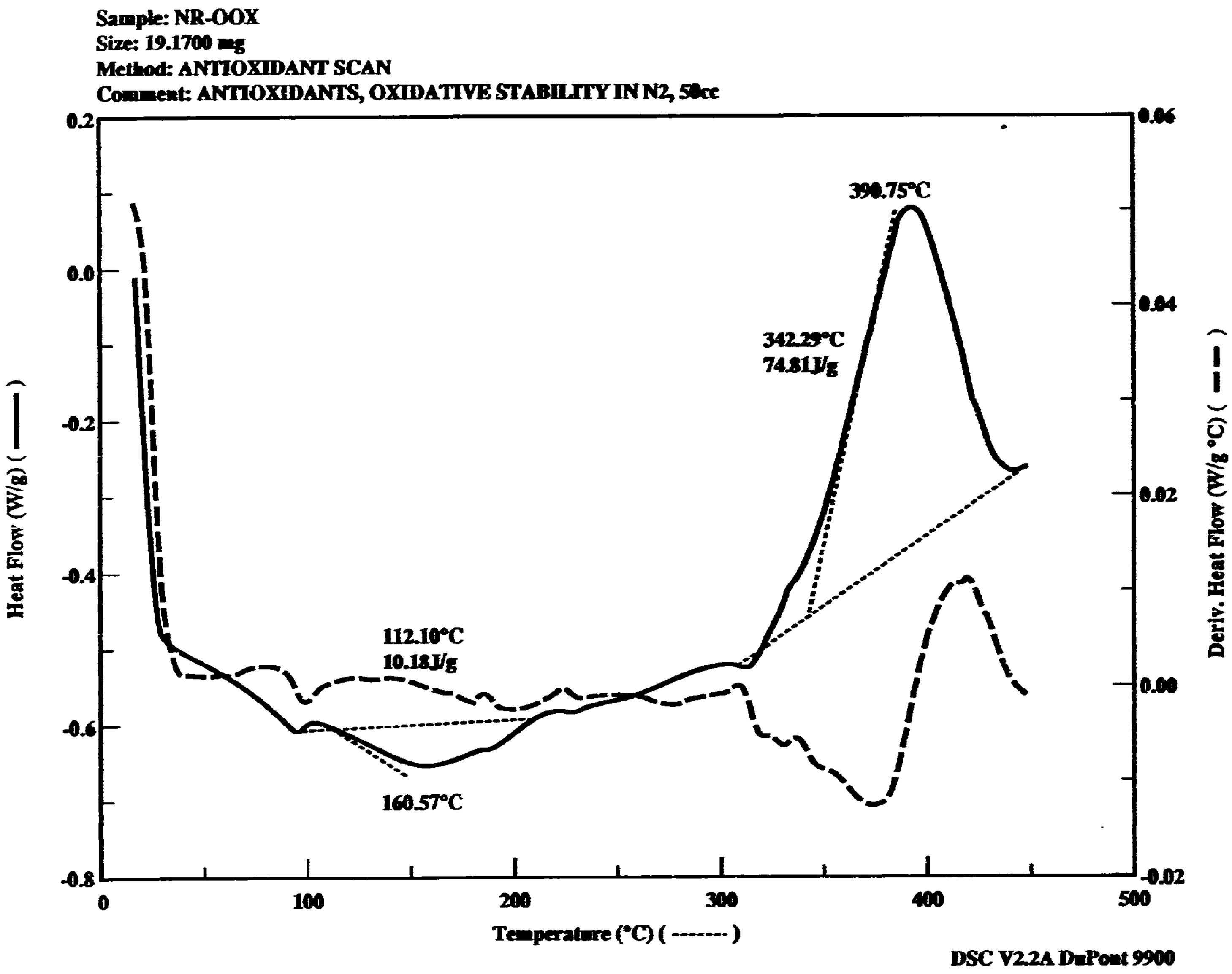


Figure A-43. DSC, NR-OOX

Sample: NR-GGX
Size: 18.9500 mg
Method: ANTIOXIDANT SCAN
Comment: ANTIOXIDANT OXIDATIVE STABILITY IN N2, 50cc/Min

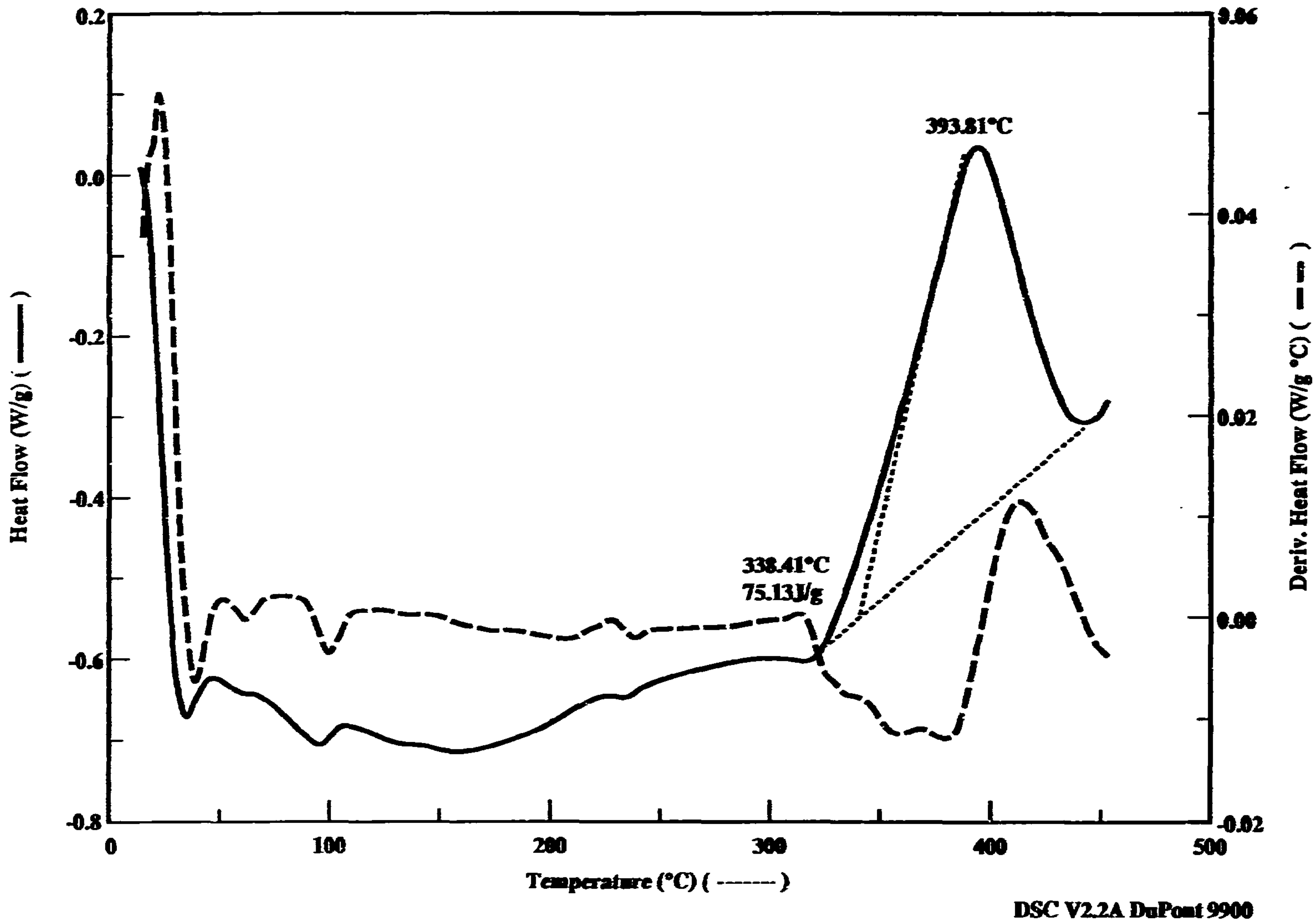


Figure A-44. DSC, NR-GGX

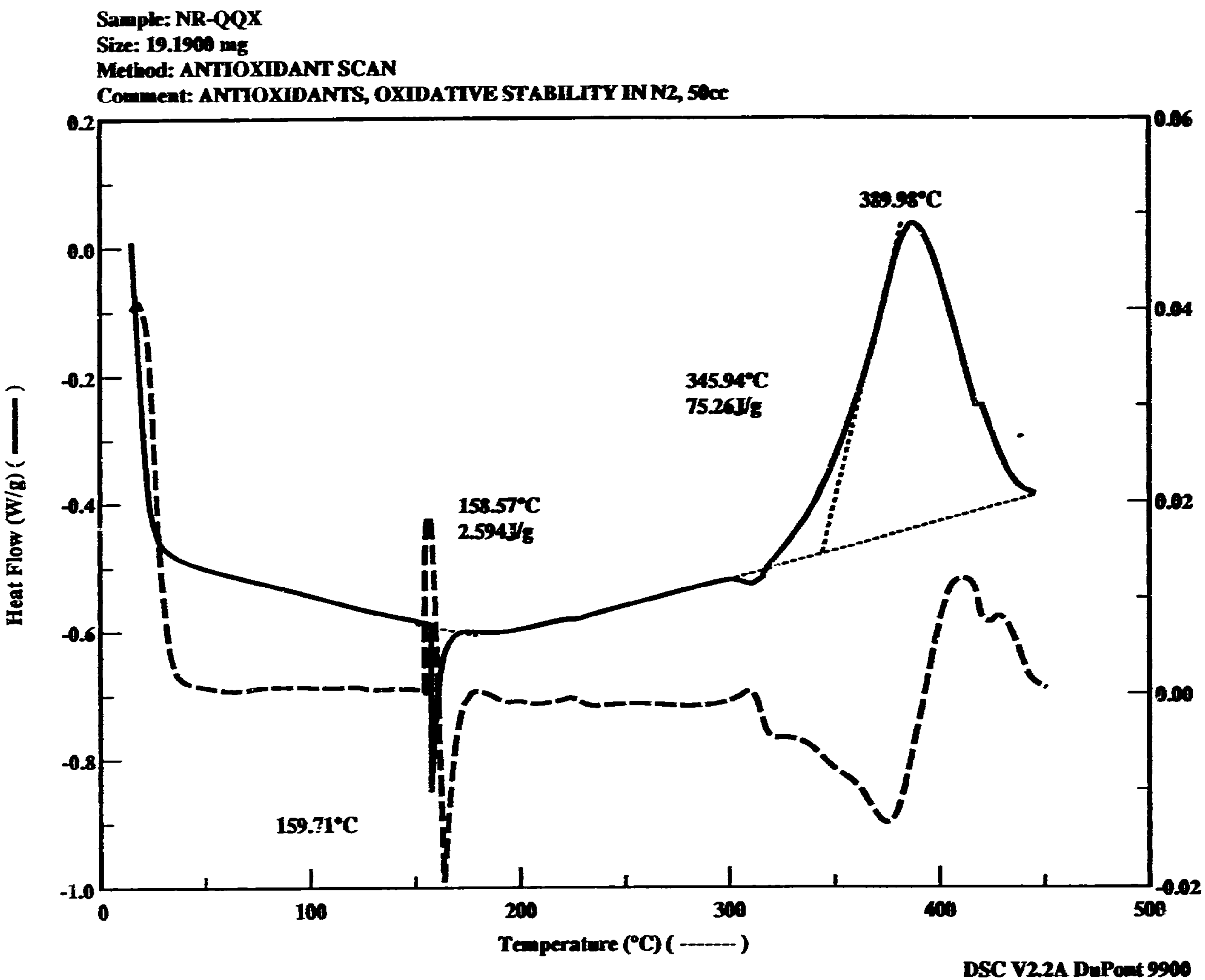


Figure A-45. DSC, NR-QQX

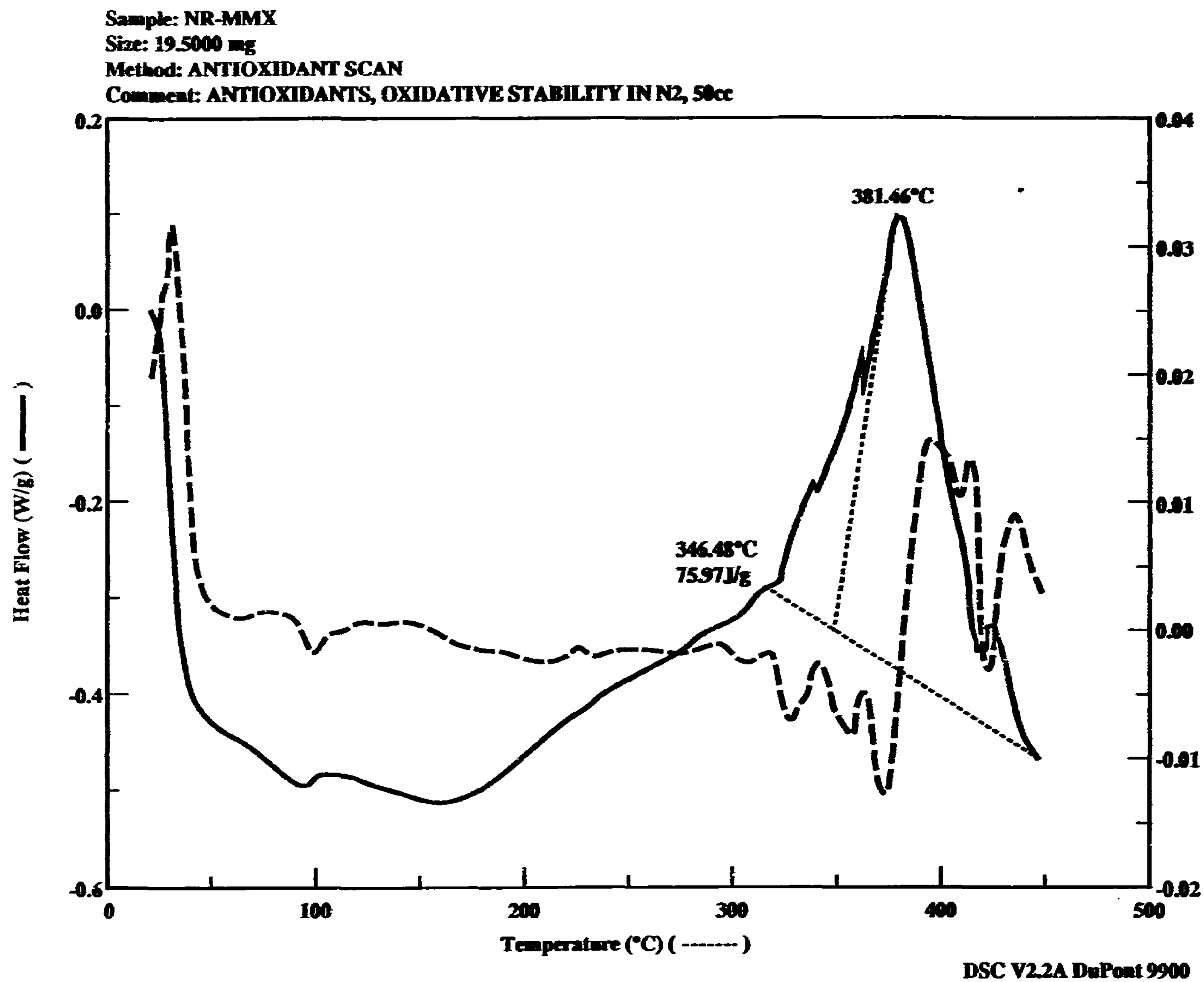


Figure A-46. DSC, NR-MMX

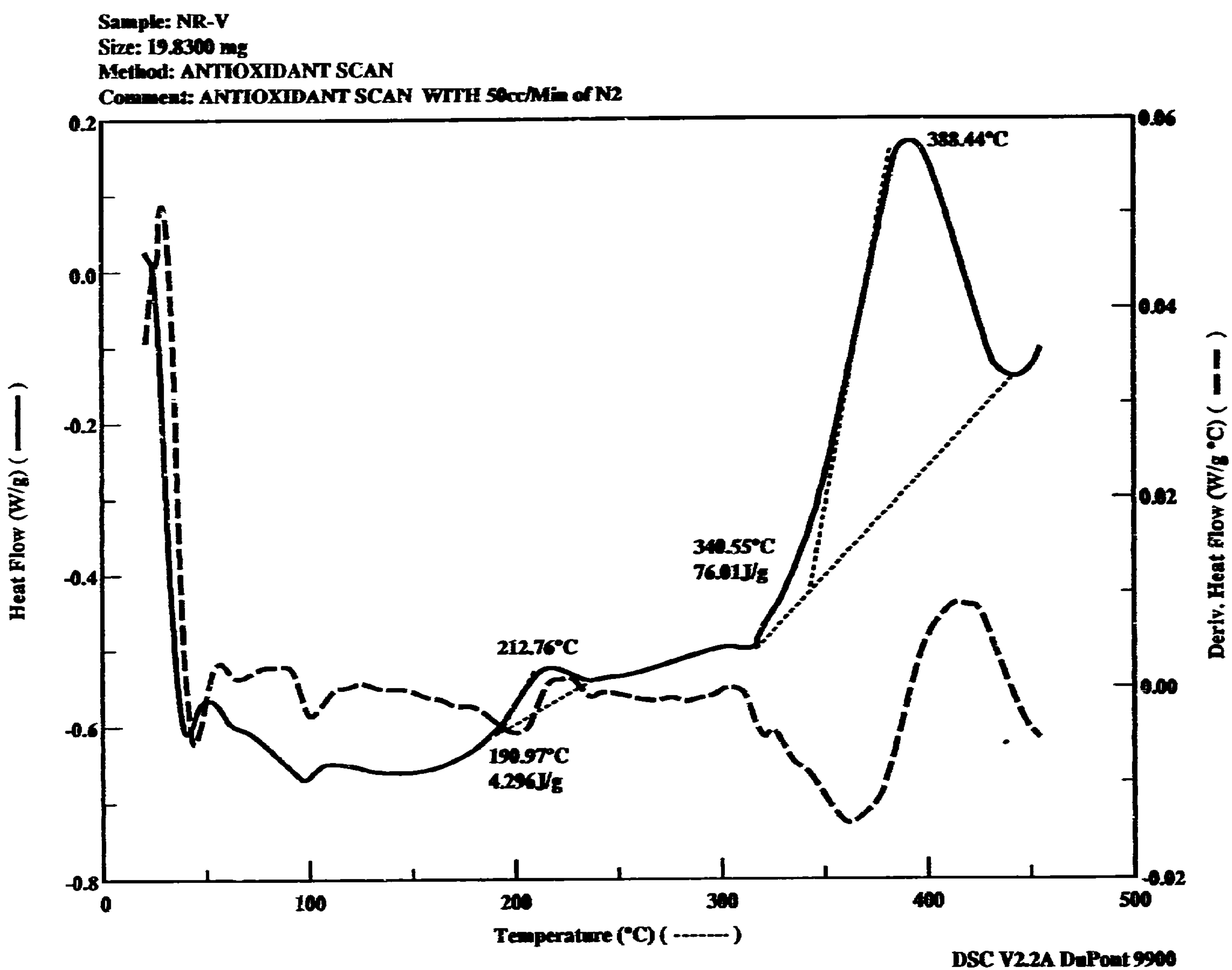


Figure A-47. DSC, NR-V

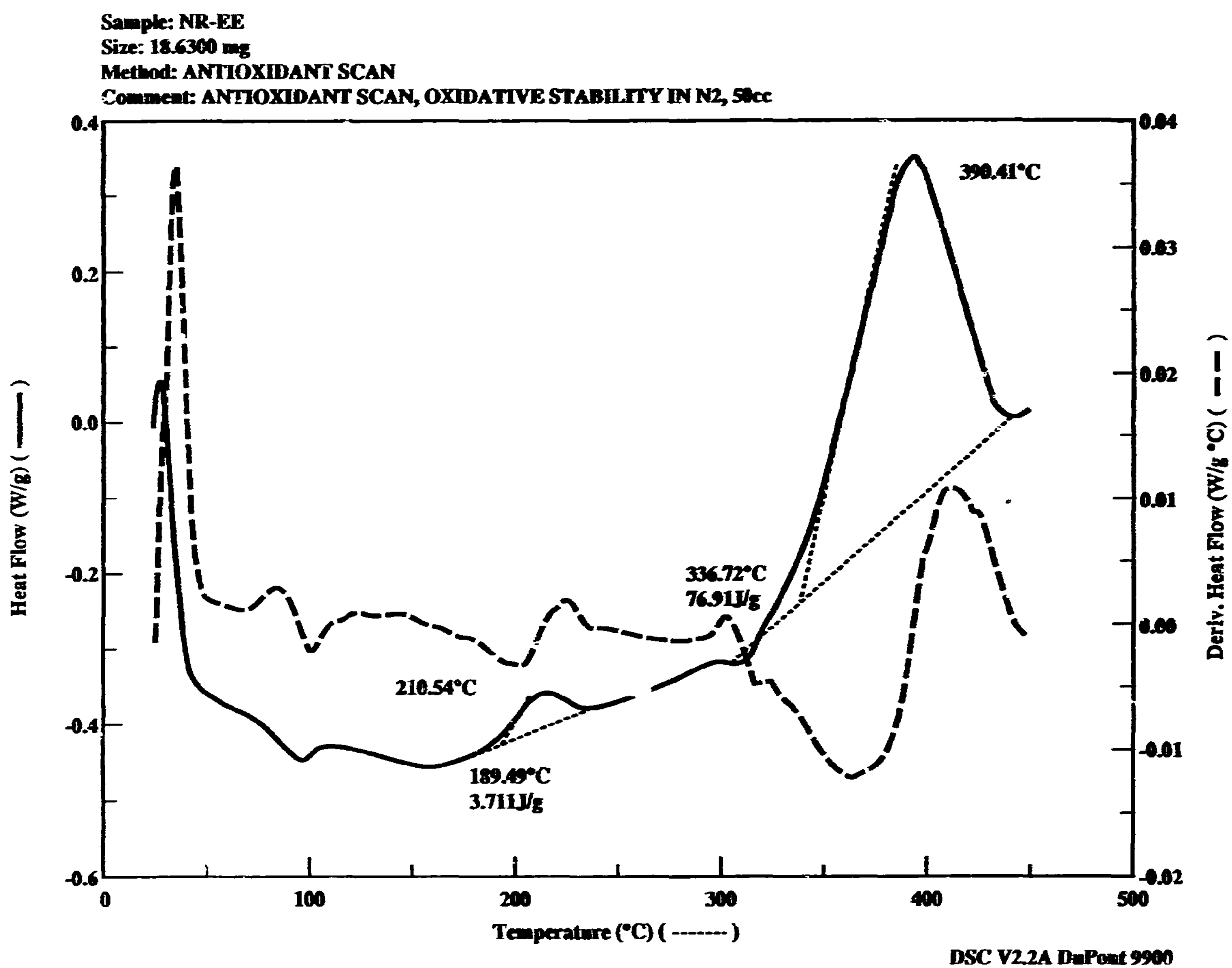


Figure A-48. DSC, NR-EE

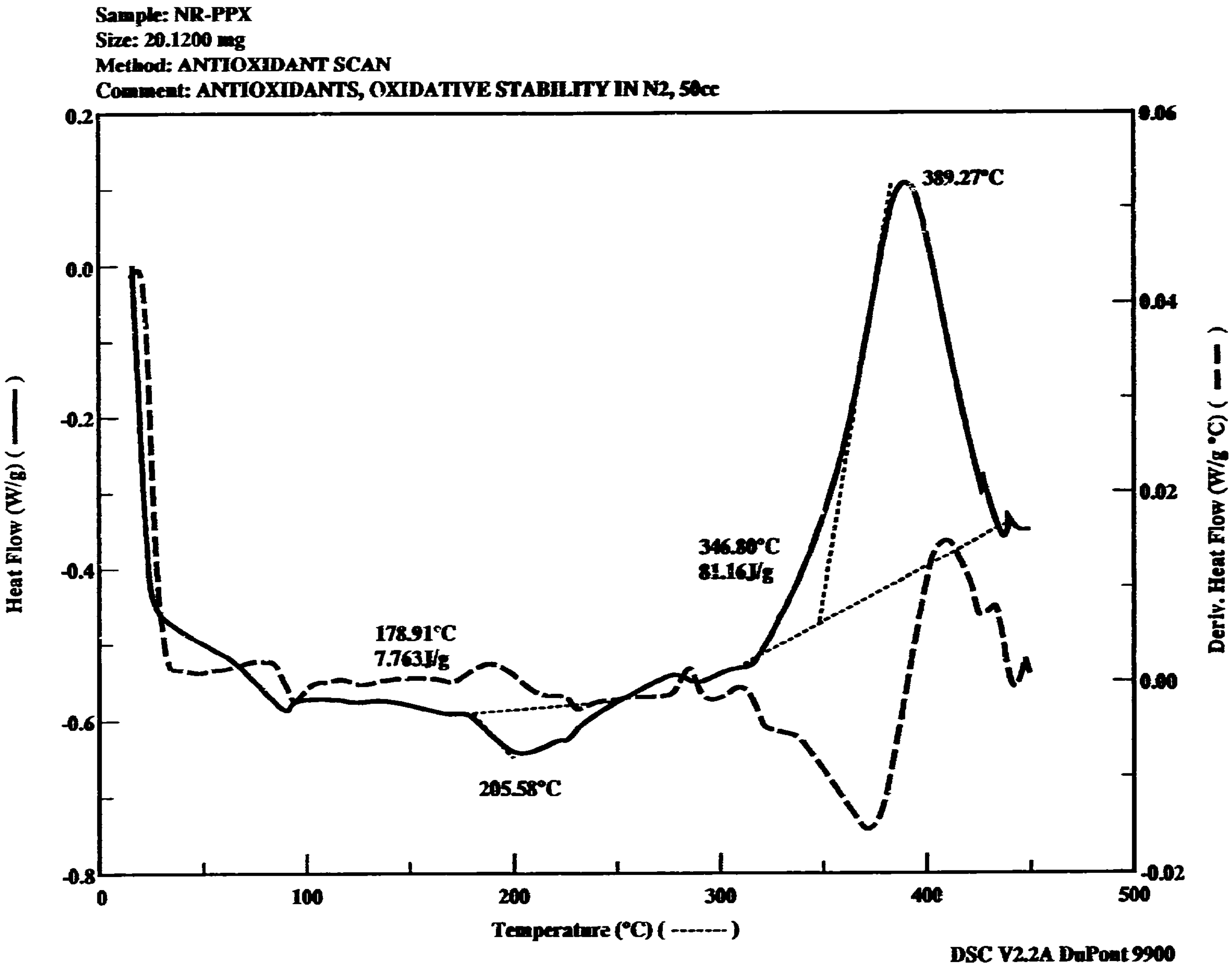


Figure A-49. DSC, NR-PPX

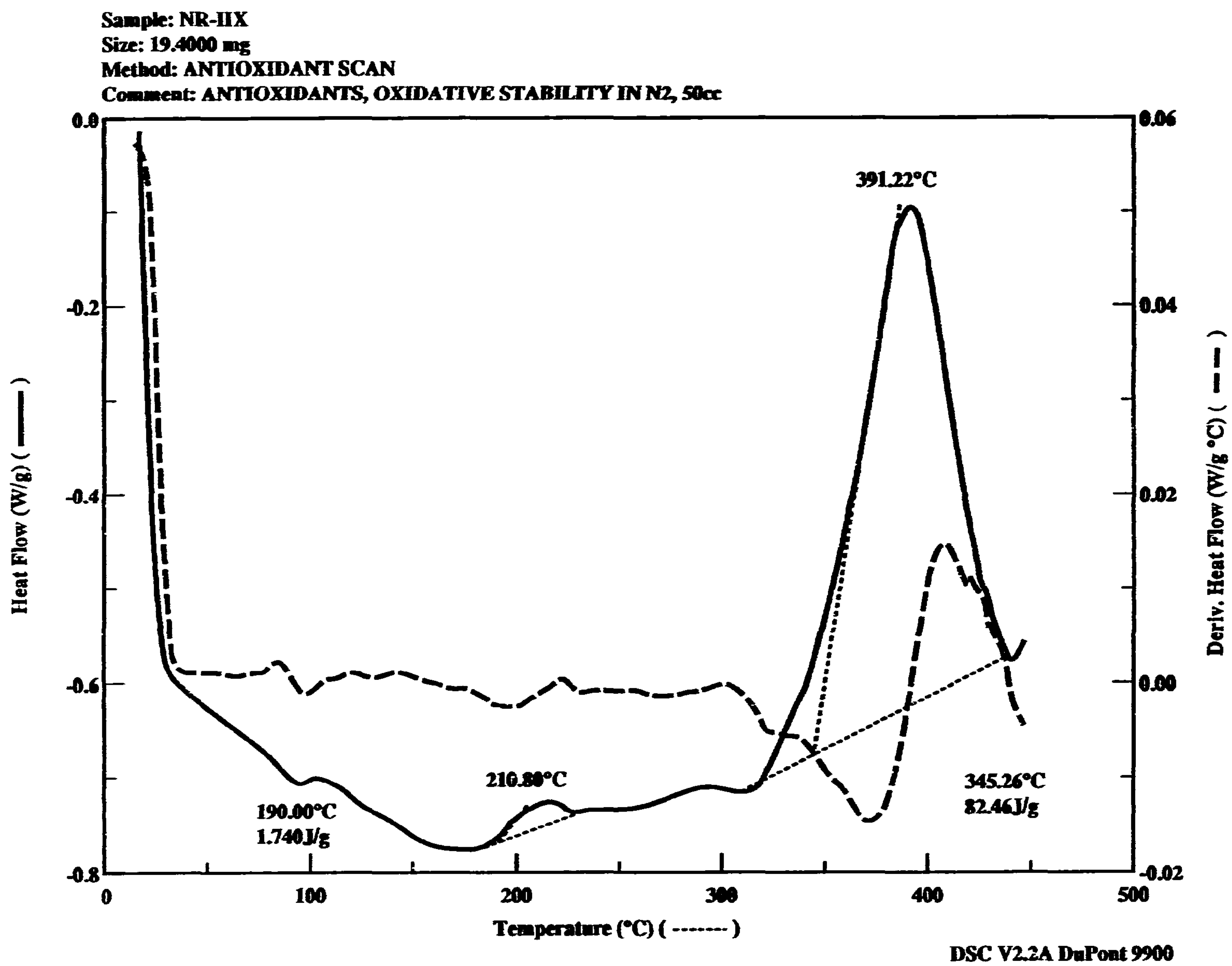


Figure A-50. DSC, NR-IIX

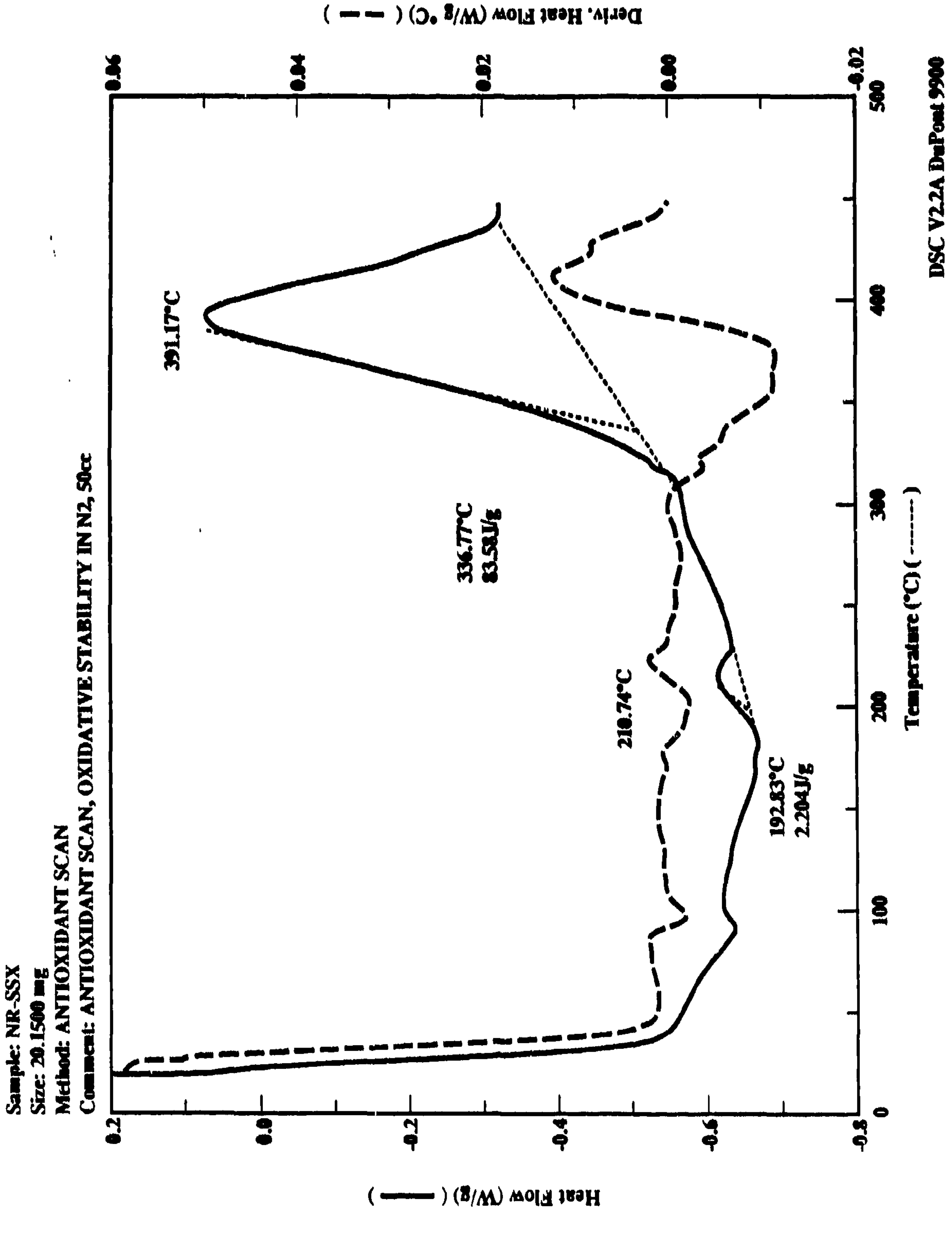


Figure A-51. DSC, NR-SSX

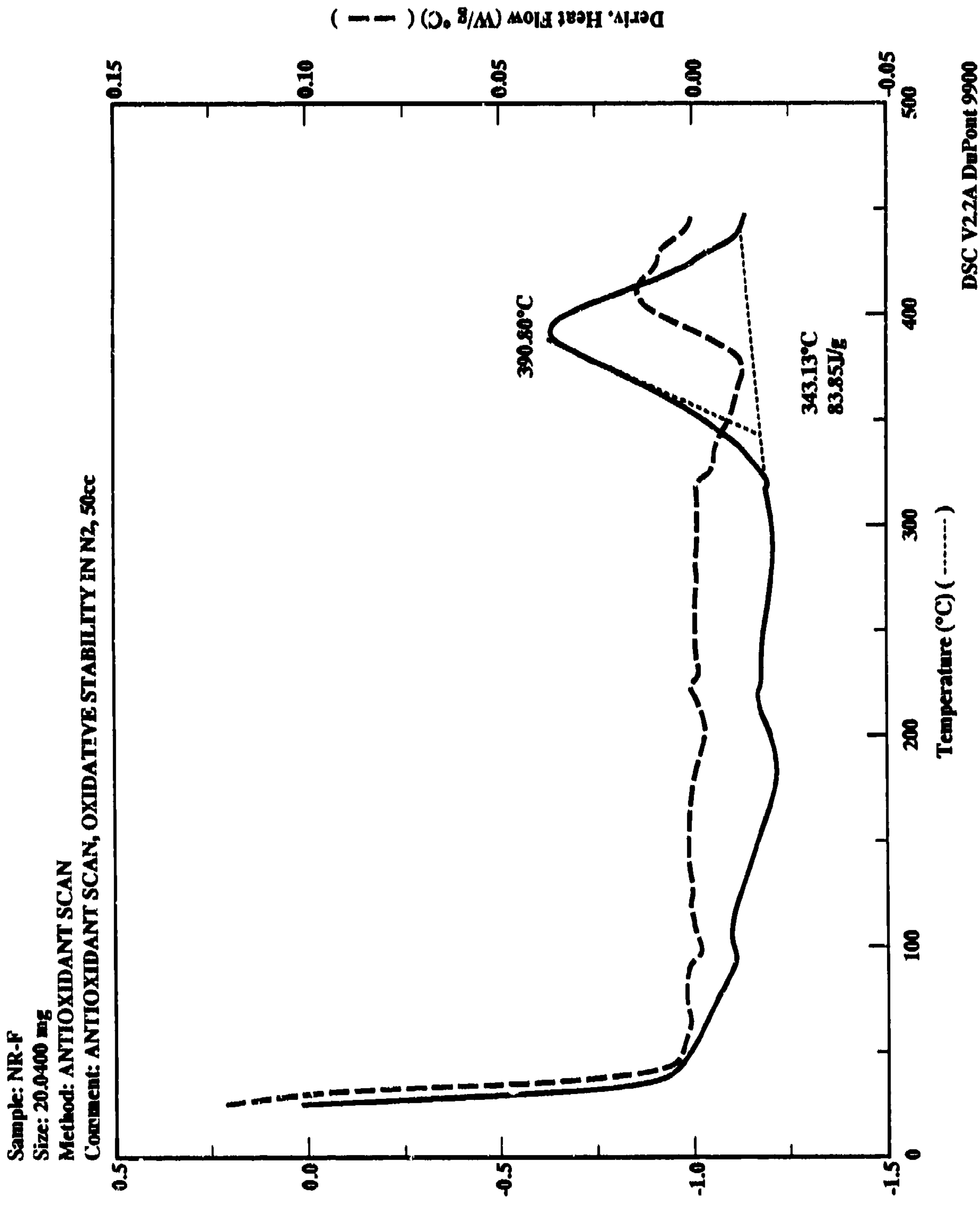


Figure A-52. DSC, NR-F

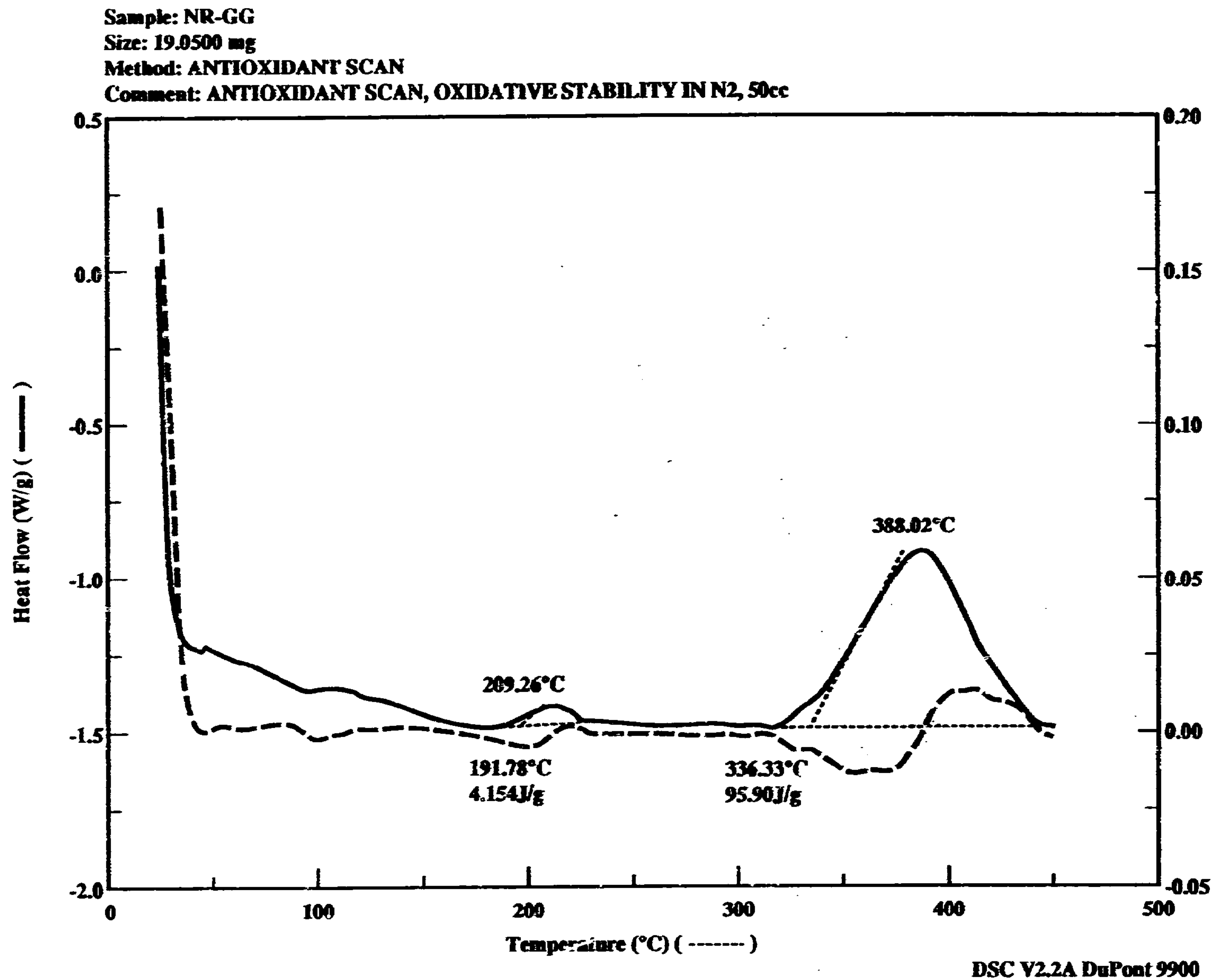


Figure A-53. DSC, NR-GG

Sample: NR-FF
Size: 19.9400 mg
Method: ANTIOXIDANT SCAN
Comment: ANTIOXIDANT SCAN, OXIDATIVE STABILITY IN N2, 50cc

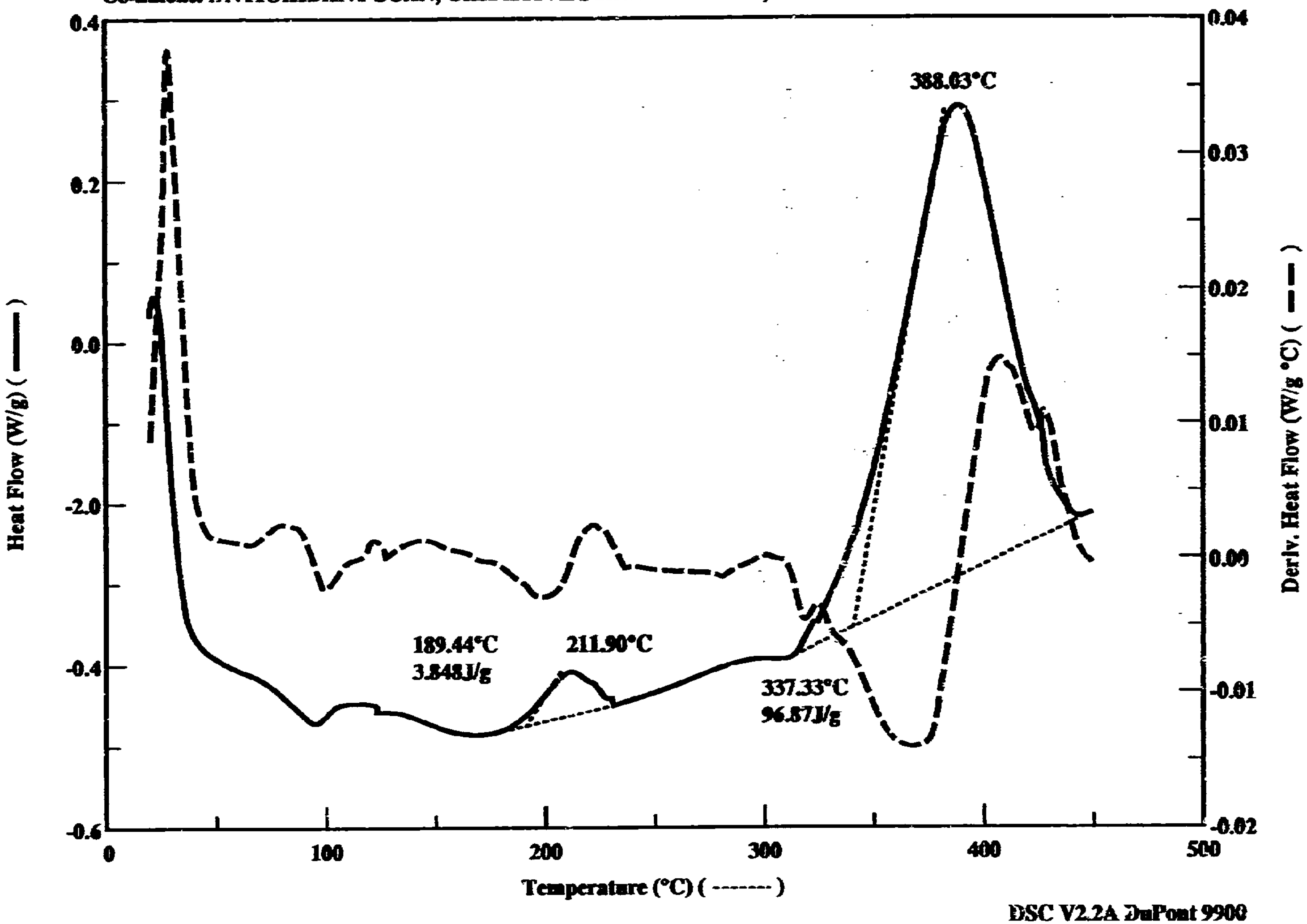


Figure A-54. DSC, NR-FF

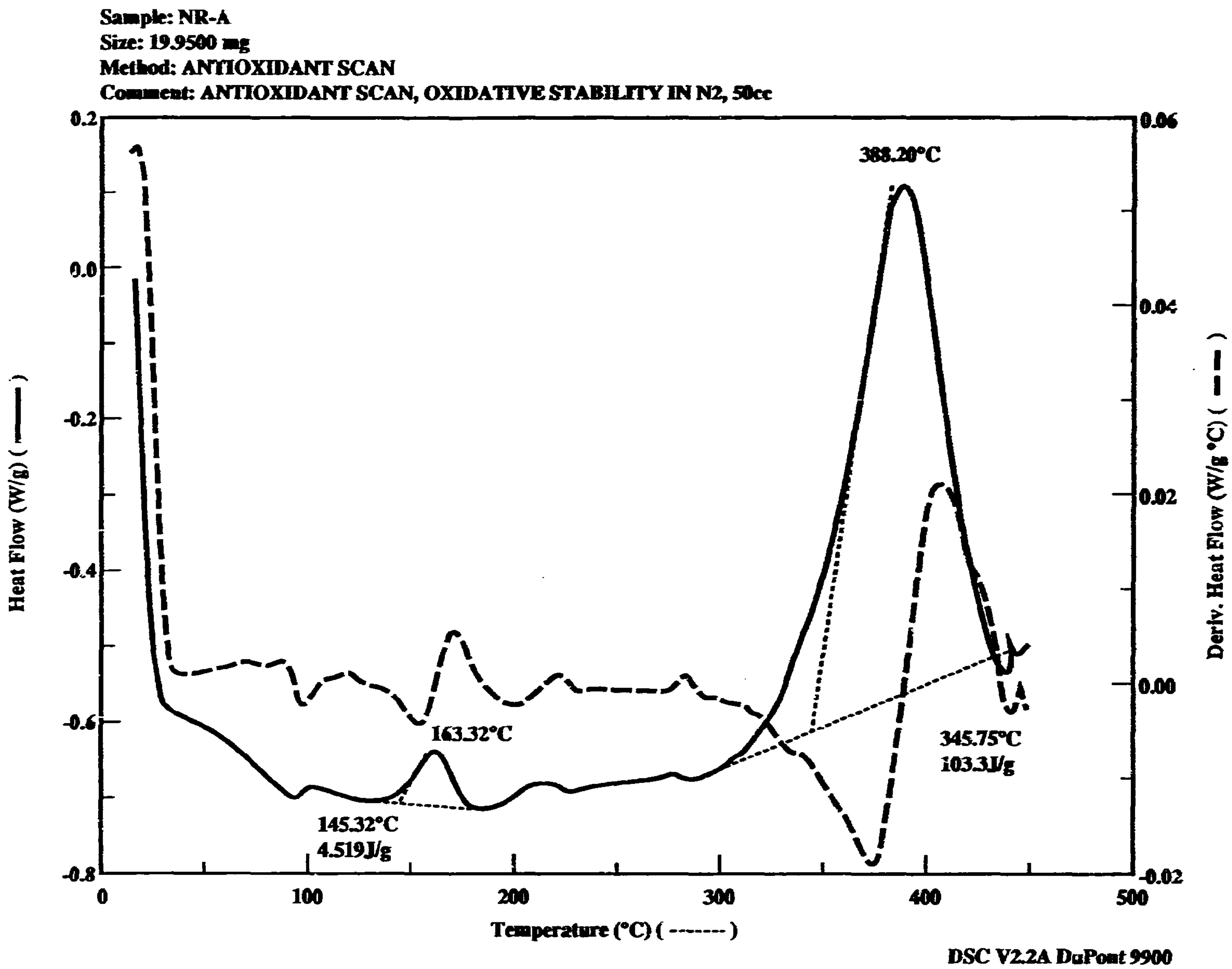


Figure A-55. DSC, NR-A

Sample: NR-NNX
Size: 19.5100 mg
Method: ANTIOXIDANT SCAN
Comment: ANTIOXIDANT SCAN, OXIDATIVE STABILITY IN N2, 50cc

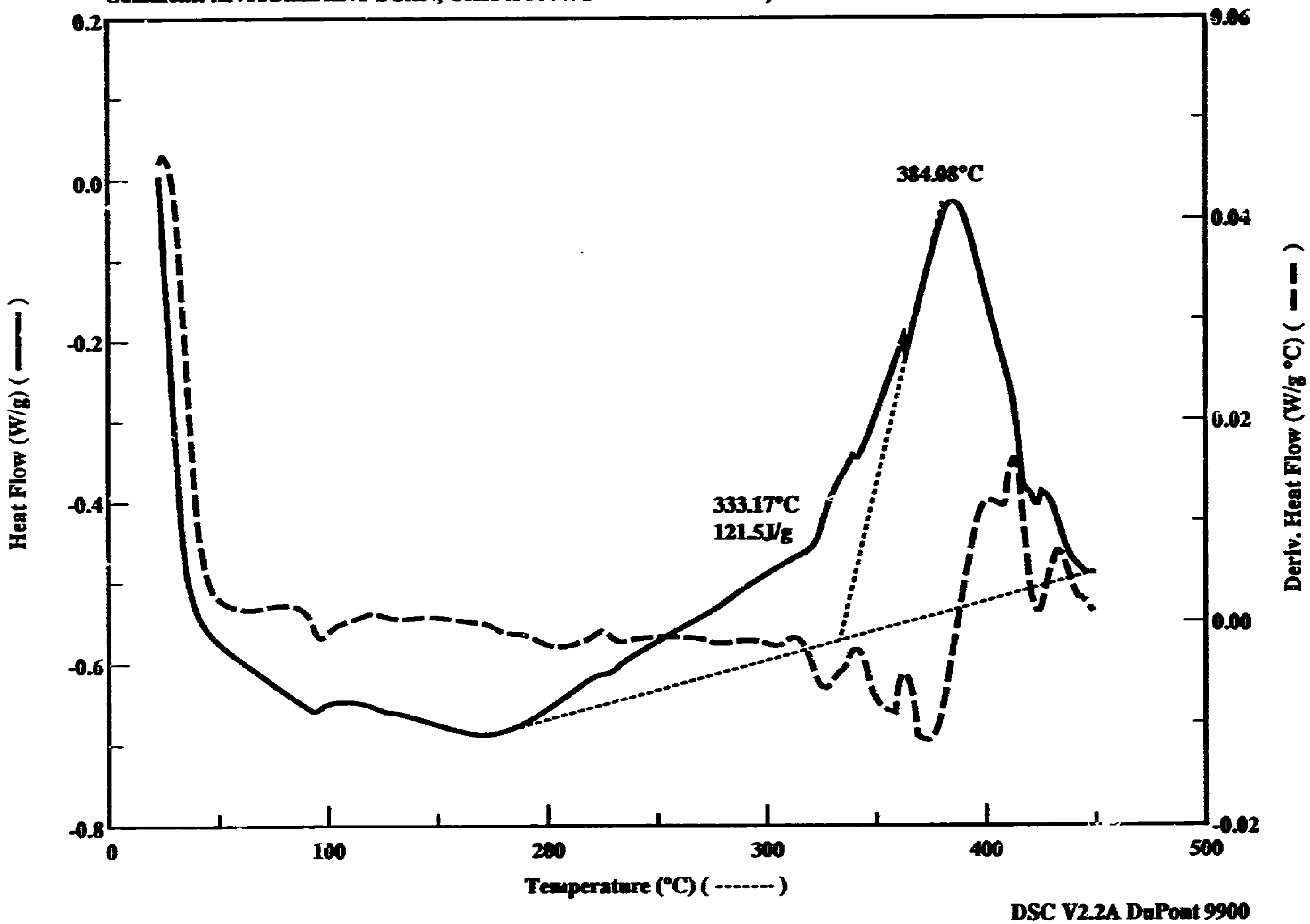


Figure A-56. DSC, NR-NNX

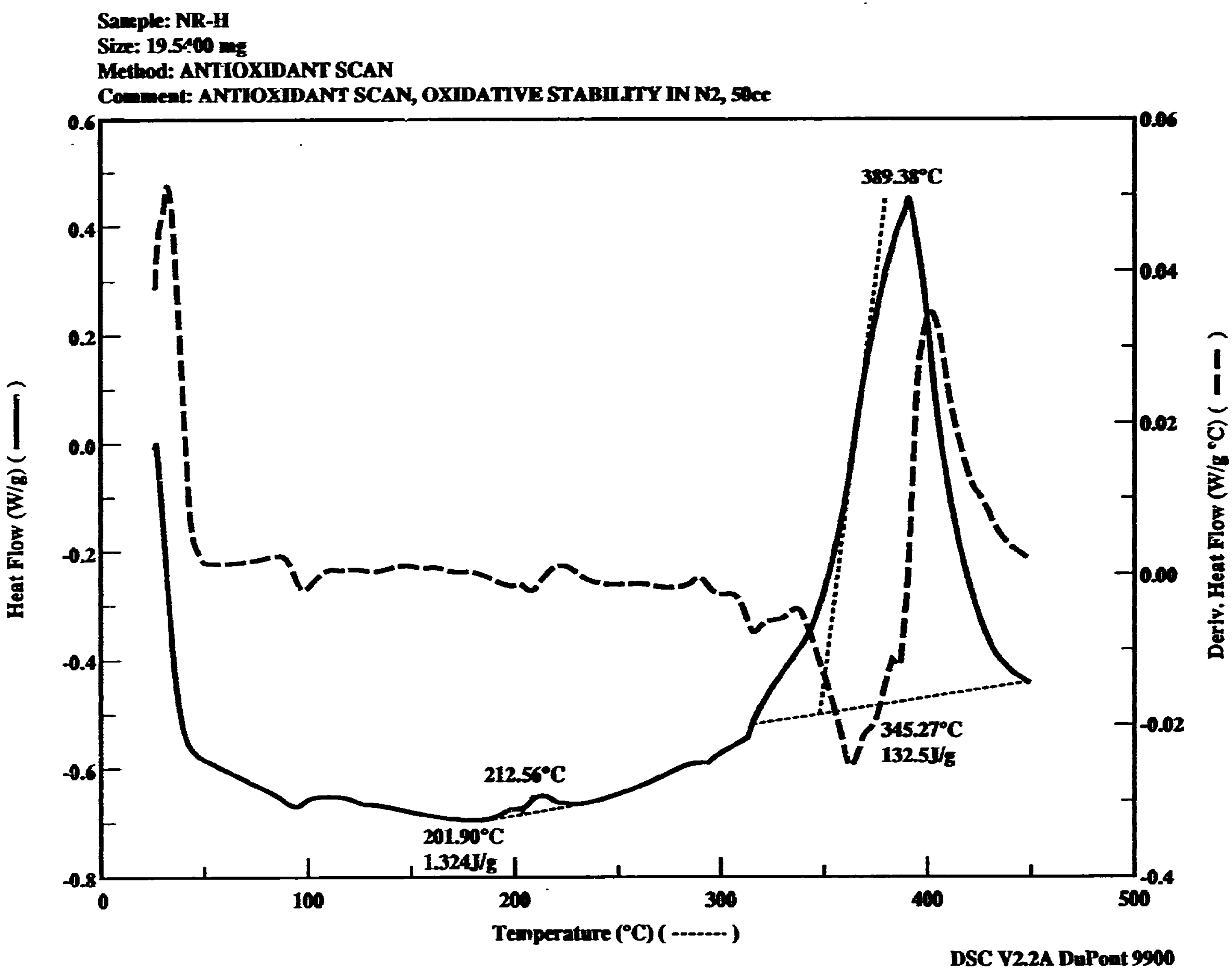


Figure A-57. DSC, NR-H

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